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DISCONNECT COUPLING REQUIREMENTS
FOR FLUORINE CONTAINING OXIDIZERS
(U)

P.W. Van Horn
Douglas Aircraft Company, Inc.

Technical Report AFRPL-TR-66-89
DOUGLAS REPORT DAC-59241
JUNE 1966

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FOREWORD

(U) This is the Phase I report on the Disconnect Coupling Requirements for Fluorine Containing Oxidizers Program, performed by the Douglas Aircraft Company, Inc., for the Air Force Rocket Propulsion Laboratory under Contract AF-04(611)-10798, dated 3 May 1965. The period of performance covered by this report is from 1 July 1965 to 1 March 1966. Mr. J. R. Lawrence (RPRP) was AFRPL Project Engineer.

(U) Douglas personnel who contributed to the reported effort are Mr. G.F. Doel, Mr. W.D. English, Mr. A.R. Rowswell, Mr. H.H. Spieth, and Mr. P.W. Van Horn, who served as Project Engineer.

(U) The report has been given Douglas Report No. DAC 59241. It has been reviewed and is approved.

UNCLASSIFIED ABSTRACT

The Phase I effort described here has resulted in a set of preliminary criteria for quick disconnect (QD) couplings for use with fluorine and fluorine containing oxidizers. For this study a number of vehicle systems were examined to determine the fundamental requirements for couplings. Also, data were obtained from literature, government and industry contacts, and Douglas studies. Identified are the requirements imposed on a typical upper stage oxidizer fill-and-drain QD by vehicle and AGE considerations, disconnect technology capabilities, specific information voids identified during this study, and program plans for Phases II and III.

The criteria established suggest that the design, fabrication, and testing of a fluorine QD can be undertaken with great confidence that the resulting product will serve its intended purpose.

Phases II and III provide for demonstrations of these criteria by fabrication and testing of two fluorine test model QD's, establishment of a preliminary military specification for a fluorine QD, and delivery of a prototype fluorine QD.

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Section I INTRODUCTION

(U) The awareness of the potential of high-energy propellants for use in advanced propulsion systems has stimulated considerable interest in the possible application of fluorine or oxidizers containing fluorine. Such use would require the establishment of design criteria for use in the design of vehicle support hardware. Of particular significance is the vehicle-to-ground oxidizer servicing line interface connector, which is usually a quick-disconnect (QD) coupling, because its design criteria are dictated by both vehicle and AGE requirements and considerations. These criteria also directly influence both vehicle and support equipment design.

(U) This has prompted the Air Force Rocket Propulsion Laboratory to initiate a three-phase program to (1) define criteria, (2) design, develop, and test a test coupling, and (3) fabricate and deliver a prototype coupling for use with fluorine. The Air Force indicated its intent in the following paragraphs from Exhibit A of the contract*:

This effort is intended to provide criteria to support the servicing requirements for future military space vehicle propulsion systems as well as existing weapon systems considered for upgrading for use with fluorine-containing oxidizers, and establish design criteria for quick disconnects for use with fluorine, fluorine compounds, or mixtures containing fluorine

(U) The Requirements Section of the contract contains the following:

"The couplings to be designed and fabricated in this procurement are intended for use at a launch complex and will be used to connect an AGE propellant servicing line to a line on the vehicle for loading or unloading propellant. This will consist of a flight assembly which will be mounted as an integral part of the vehicle structure and an AGE assembly which will be a part of the servicing system.

(U) The high toxicity and reactivity of these oxidizers imposed the most severe and restrictive requirements on the coupling: low leakage and the use of compatible materials. The last requirement all but eliminates the use of nonmetallics as primary sealing and valve seat materials. Further, these reactive and toxic characteristics demand that the device be extremely effective and be reliable in its sealing mode of operation. On the other

*Contract AF 04(611)-10798, dated 3 May 1965.

hand, the possibility of such requirements as misalignment, remote connection, and so on, increase coupling complication and reduce inherent reliability.

(U) The vehicle system selected to provide a model upon which to establish coupling criteria is MANSAT². The approach adopted identifies the requirements imposed on the MANSAT QD by vehicle and AGE considerations, and simultaneously identifies disconnect technology capabilities. These requirements and capabilities are reviewed to formulate MANSAT criteria and to identify information voids. Voids specifically identified at the beginning of the program are investigated, and definitive criteria are generated, where possible. In other cases it is only possible to identify voids, make an assessment of the potential effects they could have on the established criteria and to suggest appropriate activities for gathering needed information.

(U) In Phase I a number of vehicle systems were examined to determine the fundamental requirements for couplings and the methods used to fulfill these requirements. Data for the Phase-I effort were obtained from literature, government and industry contacts, and from studies and investigations conducted at Douglas. Industry and governmental contacts were made, for the most part, without the necessity of travel; local contacts and telephone conversations yielded the bulk of the required information.

(U) One trip was necessary to obtain realistic information on launch facility regulations, toxicity imposed requirements, and toxic material dispersion phenomenon and prediction. These data were necessary for the formulation of appropriate criteria for one of the two most critical and controlling considerations on the use of fluorine and fluorine containing oxidizers in MANSAT and thus on allowable leakage for the coupling.

(U) The design criteria thus established and documented in this Phase I report are for the specific requirements imposed by the MANSAT vehicle concept as now understood. Further, the criteria have, wherever possible, allowed for maximum flexibility in design and operation, but under no conditions were they allowed to be detrimental to the MANSAT concept.

(U) Pertinent findings of this Phase-I effort are summarized in Section IV. Recommendations for the demonstration of this criteria are contained in Section V.

(C) ²MANSAT is a theoretical high-energy (LF₂/LH₂) upper stage vehicle under study by the Air Force Rocket Propulsion Laboratory and several of its contractors.

Section II

DISCONNECT REQUIREMENTS

(II) Quick disconnect couplings (QD's) are separable connectors for use at the Vehicle/AGE interface. They are likely to be used in hydraulic, pneumatic, propellant, and electrical systems when any of the following conditions are required:

Rapid connecting or disconnecting.

Remote connecting or disconnecting.

Breakaway connections.

1. OXIDIZER QUICK DISCONNECT ELEMENTS

(U) A necessary step in the process of establishing design criteria for an oxidizer QD is the identification of tentative elements and the definition of the purpose and probable requirements for each. If problem areas exist, they must be identified and effort expended to find suitable solutions. The most probable QD subcomponents are presented in the following paragraphs.

a. Bodies

(U) A QD necessarily consists of two halves. The body of each is the shell that performs the functions of containing the fluid at its operating pressure, carrying loads imposed by its attached piping, and providing a mount to the supporting structure. Consideration should be given to making the vehicle half the simpler and lighter weight portion when used to connect a servicing line to a flight vehicle. Other primary considerations for the body are its internal and external environment; for extreme temperature applications, the effect of temperature on the physical properties and dimensional changes of the material must be taken into account.

b. Interface Seal

(U) The usual function of the interface seal is to prevent escape of internal fluid to the external surroundings. However, should the system operate with an internal pressure lower than the external pressure, or if the high-pressure potential alternates between the inside and outside of the system, it may be necessary to provide a

seal that performs its function in the reverse direction or in both directions. The allowable leakage past the interface seal depends primarily on the type of fluid used in the system. For nonreactive fluids, such as air or GN_2 , substantial leakage may be permissible. However, for highly toxic and reactive fluids, such as F_2 , a near-perfect seal is essential. Limits of F_2 leakage are discussed in Section II, 4. An F_2 oxidizer system interface seal further requires that it be easily cleaned, inspected, and replaced. A multiple cycle life for the seal is highly desirable, but not necessarily an absolutely essential requirement.

c. Latching Mechanism

(U) The normal functions of the latching mechanism are to hold the two QD halves together, to supply the sealing load, to withstand external loads transmitted through the piping, and to separate reliably to allow the coupling to disconnect. On QD's that contain shutoff valves, the latching mechanism is sometimes utilized to hold the valves open. The latching mechanism may be operated manually or by remote control for either the connect or disconnect mode. Existing manually operated QD's usually utilize screw threads, ball locks, or collet type locks for latching. Remotely actuated couplings often use linear actuators; screw-thread connections cannot be conveniently used. Ball locks, collets, and toggle locks are adaptable to remote operation.

(U) For application on a space vehicle using a highly reactive fluid such as F_2 , it appears highly desirable to be able to disconnect the QD by remote control. Remote disconnecting has been demonstrated to be feasible on the Saturn IV-B and the Centaur vehicles. Remote connection of a QD on a space vehicle application is much more difficult because of the three dimensional space problem of bringing the two coupling halves together when considering the relative motions of the structures supporting the coupling halves during the mating process. High-speed operation and protection from the fluid usually are not required during connection. Remote actuation requirements are discussed further in Section II, 2. Five possible latching schemes for providing manual connecting and remote disconnecting for an F_2 QD are presented in Section II, 3. The basic requirements for a latching mechanism for an F_2 QD are that it should be simple, reliable, and add minimum weight to the vehicle.

d. Valves

(U) Valves are not a necessary requirement in a QD, but they may be included in one or both halves of the coupling. When included, they are used to help prevent entry of contamination into the system and to prevent fluid spillage or leakage when the coupling is disconnected or separated. Generally, the valves are

spring loaded closed when the coupling is separated and are designed so that the actual mating and latching of the coupling opens them and holds them open. For special applications, the valves may be provided with an independent actuation system and may be used to control the flow through the QD. The basic requirements for valves are that they should operate reliably, be made of compatible materials, have minimum pressure drop under flow conditions, and provide a seal with tolerable leakage for the application. Design considerations for this type of valve are discussed in Section II, 5.

e. Actuators

(U) Actuators are used when remote control of connecting or disconnecting of the valves is required. For an application on a servicing line to a space vehicle, the actuation mechanism would normally be included in the AGE half of the QD to minimize vehicle weight. The actuator can be designed as an integral part of the QD; it can be component mounted on the exterior of the QD; or it can be a component remotely mounted and connected to the QD with a linkage or cable system. Power to drive the actuator can be extracted from any available power source -- pneumatic, hydraulic, electric, or chemical (including explosive). The basic requirements for an actuator are that it should be simple and operate reliably.

(U) For use in remote separation of a fluorine QD, redundant actuators should be utilized. A pneumatic actuator would probably be a first choice for use with a fluorine system because it could be designed so that no incompatible materials would be introduced in the vicinity of the fluorine system. If an explosive device is used, it should be designed so that all products of operation are contained within it.

f. Purge, Vent, and Drain System

(U) A purge, vent, and drain (PV&D) system is not a general requirement for QD's. However, for special applications, such as for use with toxic or reactive fluids, it may be required. There are several functions that can be performed with a PV&D system. It can be used for disposal of leakage past the interface seal. For a coupling utilizing valves in both halves, the PV&D system can be used for extraction of trapped air from the coupling separation cavity prior to flow and for extraction of trapped fluid from the same cavity after flow. Where the QD contains no integral valves and the primary shutoff valves are downstream of the QD, it will be desirable to use a PV&D system to expel the propellant from appropriate portions of the transfer system that contains the QD. Methods of performing this task are discussed in Sections II, 2 and II, 11. The PV&D system also may be used to detect leakage past the primary valves prior to QD separation. The basic requirement for a PV&D system for use in connection with a fluorine QD

is that it be made of fluorine-compatible materials. Fluorine-compatible shutoff valves would be required in both the purge, drain, and vent piping. The same stringent cleaning requirement applicable to the main oxidizer system also would be applicable to this system. A further requirement is that the purge fluid must be fluorine compatible and free from contamination.

g. Shroud

(U) A shroud is a shield or barrier used to provide a zone of isolation between a component and its normal environment. For the general application of QD's, it would not be required. However, for cryogenic applications it is useful, when combined with a dry gaseous purge, as a means of isolating the cold surface of the QD from atmospheric water vapor, thus preventing the buildup of ice, which could interfere with latch or valve operations. A shroud is also required with some PV&D systems. Further discussion of a specific shroud for a fluorine QD is presented in Section II, 3.

h. Bellows

(U) Bellows offer flexibility to absorb thermal expansion and contraction, installation misalignment, structure deflection, and vibration. Bellows may find application in connection with a QD in several ways. A primary application is the main fluid line, which must be flexible to provide the movement required for alignment and connection of the QD halves and for QD separation. They may be used to provide both the preload and a pressure energized loading for the interface seal and to transmit motion for valve operation without the use of sliding seals.

(U) The basic requirements for bellows when used in a fluorine oxidizer system include use of compatible materials, adequate fatigue strength at the environmental temperatures and flow conditions, and use of a shape that permits cleaning and inspection of all surfaces exposed to the oxidizer. Because of the thin material from which bellows are constructed, they are highly vulnerable to a drastic loss of strength if surface corrosion or pitting occurs; therefore, careful attention must be given to the selection of a compatible material for this element. Because fluorine and FLOX are cryogenic, it is necessary to ascertain that any bellows materials used with these oxidizers have adequate physical properties at cryogenic temperatures. Bellows used for fluorine service should always be of the open convoluted type to facilitate cleaning. Welded nested bellows are extremely difficult to clean and to inspect for cleanliness and should not be used for this application. Lap welded seams in bellows should also be avoided because of possible contaminant entrapment.

1. Leak Detection

(U) A leak detector is not a general requirement for QD's. However, because of the high toxicity and reactivity of fluorine oxidizers, a means for detecting leakage and quantitatively monitoring atmospheric contamination is necessary. The permissible limits of atmospheric contamination from a launch facility using toxic propellants are presented in Section II, 4. There are various ways in which leak detectors could be used advantageously at this type of facility. One would be to locate sensors at strategic points and to conduct a continuous or scheduled intermittent sampling of the atmosphere for F_2 contamination. For a specific component such as a QD, which because it is a separable joint may have a higher than average probability of leakage and because it is proposed to use an inert-gas purged shroud around the QD to keep atmospheric water vapor away, it may be desirable to use F_2 sensors to monitor the discharging purge gas for possible F_2 leakage through the QD interface seal and thus obtain the earliest possible indication of seal leakage. Another use for a leakage sensor is to monitor the F_2 concentration in the vent side of the QD PV&D system to determine when the oxidizer transfer line is adequately purged to permit QD separation. This same sensor could also serve as a failure indicator if the shutoff valve on either side of the QD did not close properly.

(U) The basic requirements for a leak detector are that it reliably measure F_2 concentration, have a short response time, and have a reasonably long service life. Although some work has been done with F_2 leak detectors, actual experience is limited and an adequate system for use at a launch facility will undoubtedly require additional development. Several possible F_2 leak detection devices are discussed in Section II, 10.

2. VEHICLE AND AGE REQUIREMENTS

a. General

(U) The servicing of upper stage vehicles with oxidizers containing fluorine should be conducted under favorable meteorological conditions, little or no release of the oxidizer, and under the direct control of qualified personnel. In general, the transfer equipment and techniques will be much the same as those presently used with liquid nitrogen and liquid oxygen. However, the toxic and reactive nature of fluorine severely limit material selection, require leak-tight systems, and require ducting of vented oxidizer to safe disposal areas. Oxidizer filling and draining of the vehicle can be performed through the same Vehicle/AGE interface coupling; however, separate Vehicle/AGE interface couplings will be required in the oxidizer vent system.

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(U) In establishing specific requirements for these couplings, the following must be considered: interrelations between the location of the Vehicle and AGE oxidizer shutoff and isolation valves, requirement for, and location of, redundant valving, permissible quantities of toxic vapors that may be released into the atmosphere at time of coupling release, capability of the oxidizer drain and purge systems to reduce oxidizer concentrations to acceptable limits, need for remote connection, and need for remote and/or rapid release at time of disconnect. The discussion to follow covers the important and controlling aspects of these considerations.

b. MANSAT System Studies

(C) The MANSAT Vehicle concept as now envisioned is a multiple-restart, Earth orbiting, maneuverable upper-stage vehicle that uses a high-energy fluorine/hydrogen propulsion system. Preliminary design of this propulsion system has been accomplished under separate contract, and the results are presented in Appendix I.

(U) The entire vehicle system is shown in Figure 1 to afford a concise physical frame of reference upon which to base the ensuing discussions.

(C) Propellants are fed to the engine through low-pressure ducting, as shown schematically in Figure 2. At engine shutdown, the LF_2 in the feed line is trapped between the tank-mounted valve and the engine pre valve. The LF_2 in the vicinity of the engine is vaporized as a result of engine heat feedback, and the vaporized fluorine is displaced through the bleed line into the tank to prevent excessive pressure buildup in the feed line during coast.

(C) The fill line has a shutoff valve that seals the tank prior to launch. This valve is located close to the tank wall in the highly insulated portion of the system to keep liquid out of the fill line during coast, thereby reducing heat leakage into the system and minimizing the trapping of unusable oxidizer. The umbilical propellant transfer line is connected to the vehicle fill system with a QD coupling, which does not have internal valves to trap oxidizer.

(C) Both tanks are vented during propellant filling through separate vent lines connected to the vehicle sidewalls. Incorporated in each vent line is a shutoff valve that seals the tank prior to launch. This valve is located close to the tank, thereby reducing heat leakage into the propellant. The QD coupling does not have any internal shutoff provision, thus allowing emergency venting during the mission.

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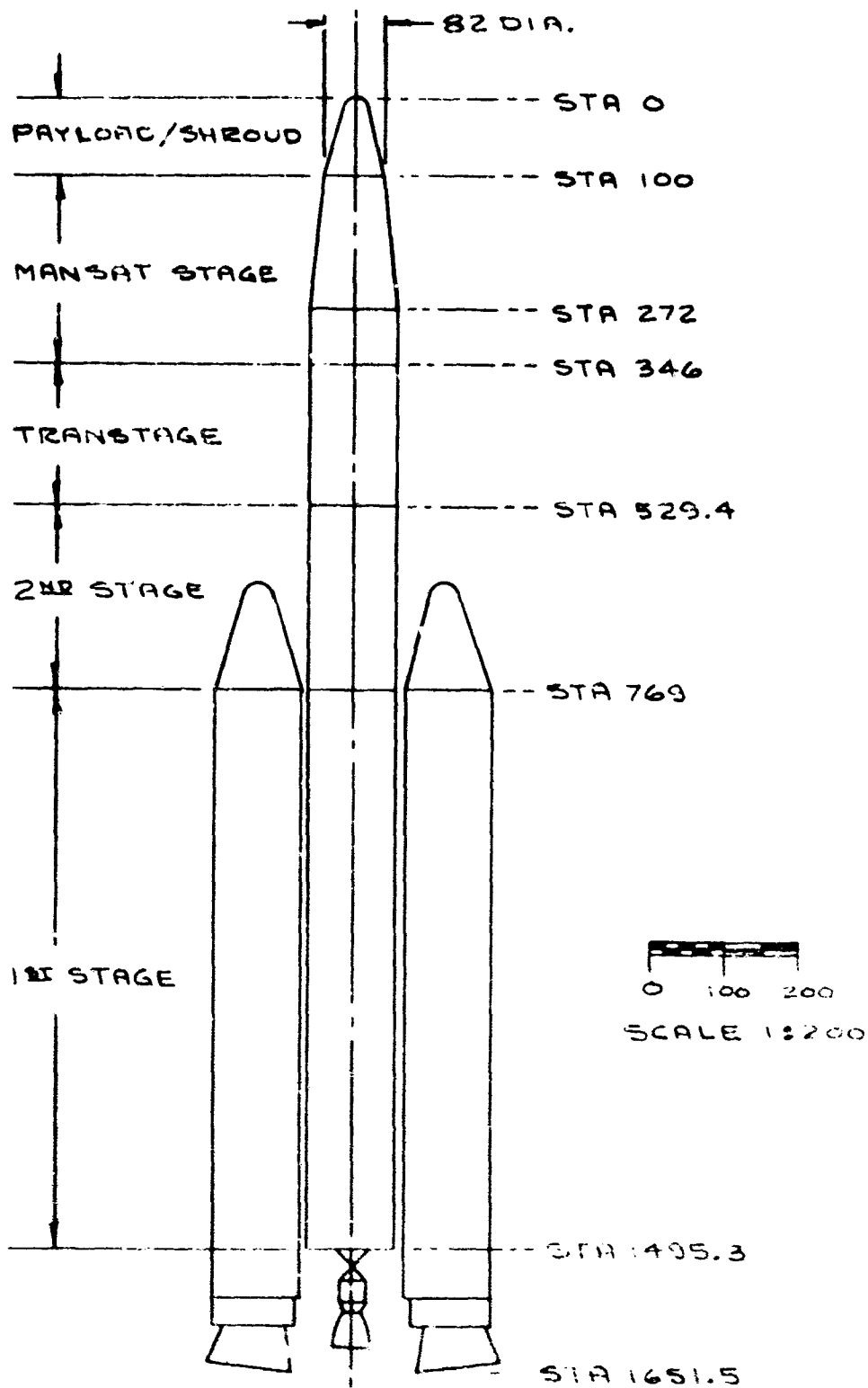


Figure 1. Titan III C/MANSAT

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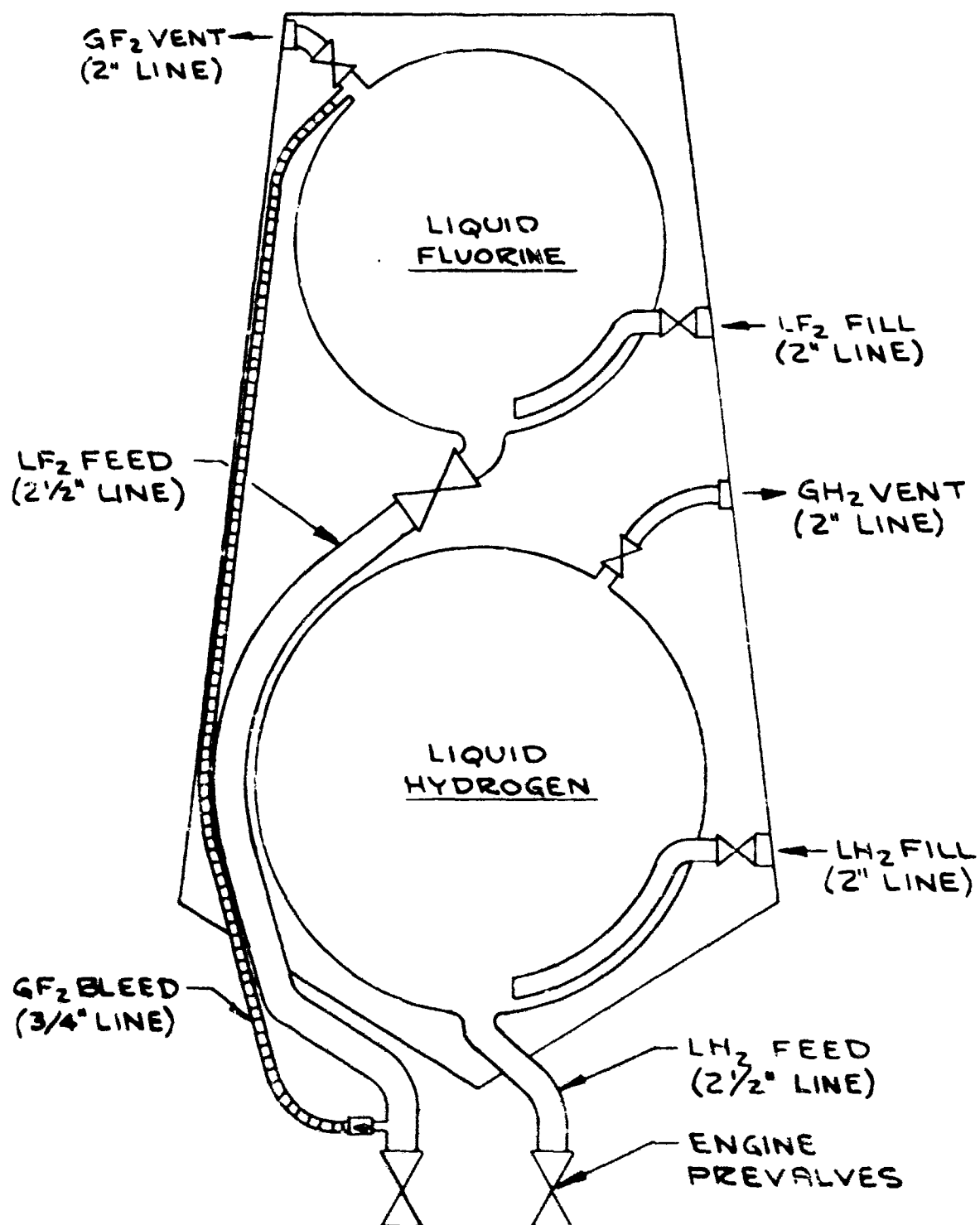


Figure 2. MANSAT Fill and Feed Schematic

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c. Draining of Liquid Oxidizers from Lines and Components

(U) Installation of lines and components in Vehicle and AGE liquid oxidizer systems should provide rapid and complete drainage of liquid under gravity-flow conditions.

(U) Where components and lines cannot be arranged for gravity-flow drainage and liquid trap points cannot be avoided, a variety of approaches are potentially applicable for scavaging the liquid. These approaches can be categorized into one of the three following classes:

- (1) Vaporization and removal as gas. Pressurization and purge gases can be effectively used to remove liquid from trapped areas in components, lines, and fittings by promoting heat and mass transfer under turbulent flow conditions, thus resulting in vaporization and gas-stream entrainment of the liquid in the purge gas.
- (2) Removal by mixing and displacement with another liquid (probably an inert and otherwise compatible fluid).
- (3) The use of special drain lines and/or fittings to promote gravity-flow drainage.

(U) The use of a liquid to mix with and displace the oxidizer has obvious drawbacks: (1) the mixture must be vented or otherwise disposed of, thereby probably requiring specialized disposal or venting equipment; (2) eventually the displacement liquid trapped in the system must be removed because its presence during a subsequent operation of the system would result in oxidizer contamination and dilution and could lead to degradation of performance and hardware integrity.

(U) A significant consideration involved in the liquid displacement approach is the type and availability of the displacement liquid. Undoubtedly, liquid nitrogen will be plentiful at liquid fluorine and liquid FLOX installations and would be a suitable purging fluid for these oxidizers if the cleanliness of the nitrogen and its transfer system can be assured. Great care must be taken to preserve the integrity of the system for oxidizer service.

(U) Liquid oxygen is a possible displacement fluid for both fluorine and FLOX. The addition of filtering and purification processes to maintain adequate system cleanliness is quite likely because oxygen systems normally contain more nonmetallic particulate than are allowable in fluorine systems. The use of oxygen further introduces another hazardous, although nontoxic, fluid.

(U) Gas-phase removal following vaporization of the liquid is straightforward. The primary problem is effecting the vaporization.

This becomes more difficult when the trapped liquid is in a highly insulated (low-heat leak or subcooled) portion of the system. To promote vaporization, deliberate introduction of the heat of vaporization from external sources may be necessary; hot purge gases may be the answer and can be applied both externally and internally.

(U) Externally applied purges usually will be far more difficult to handle because of the added complexity of having to effectively shroud the exterior of the component or line containing the trapped liquid. If these trap points are buried inside of a complex device or in highly insulated areas of the system, the external purge approach to vaporization of trapped liquid is useless.

(U) Much more effective are internally applied purges because they permit direct contact of the warmer purge gas with the liquid to be vaporized. One notable exception is dead-ended areas of the system, components, and instrumentation lines. It is desirable to force purge gases under highly turbulent flow conditions into these trap points, to cause the liquid to break up into droplets (by agitation or other suitable means), to force the mixing of these droplets with the purge gases, and to promote the rapid turnover (replacement) of purge gases in the system. Special techniques or devices may be required to adequately effect these desired results.

(U) In the vehicle fill and drain system, the line from the vehicle/AGE interface coupling to the vehicle fill and drain valve is dead-ended and will probably contain several trap points, even if slightly elevated to promote liquid drainage. These trap points are found in bellows, at component and line connection points, and in valves. Should the vehicle design be such that it is impossible to prevent trap points in the fill line, the trapped oxidizer must be purged from the system or effectively isolated and carried along as inert unusable weight. The last approach is undesirable because every pound of unusable propellant directly reduces the vehicle payload capability.

(U) If the vehicle lines are short, a high-velocity stream of purge gas directed from the ground half of the coupling might prove adequate for oxidizer removal.

(U) When the vehicle line is long, or has a large L/D, another approach must be used. Two possible vehicle fill and drain line configurations are shown in Figure 3. Configuration 1 results in trapping of liquid in the convolutes of the bellows. Removal of liquid through application of turbulent gas flow will require that the purge gas be introduced near the dead-ended portion of the line. This could be achieved by introducing the purge gas through a concentric purge line located inside the fill and drain line, as shown in Configuration 1. The purge gas would then promote the rapid and effective evaporation and removal of the trapped oxidizer.

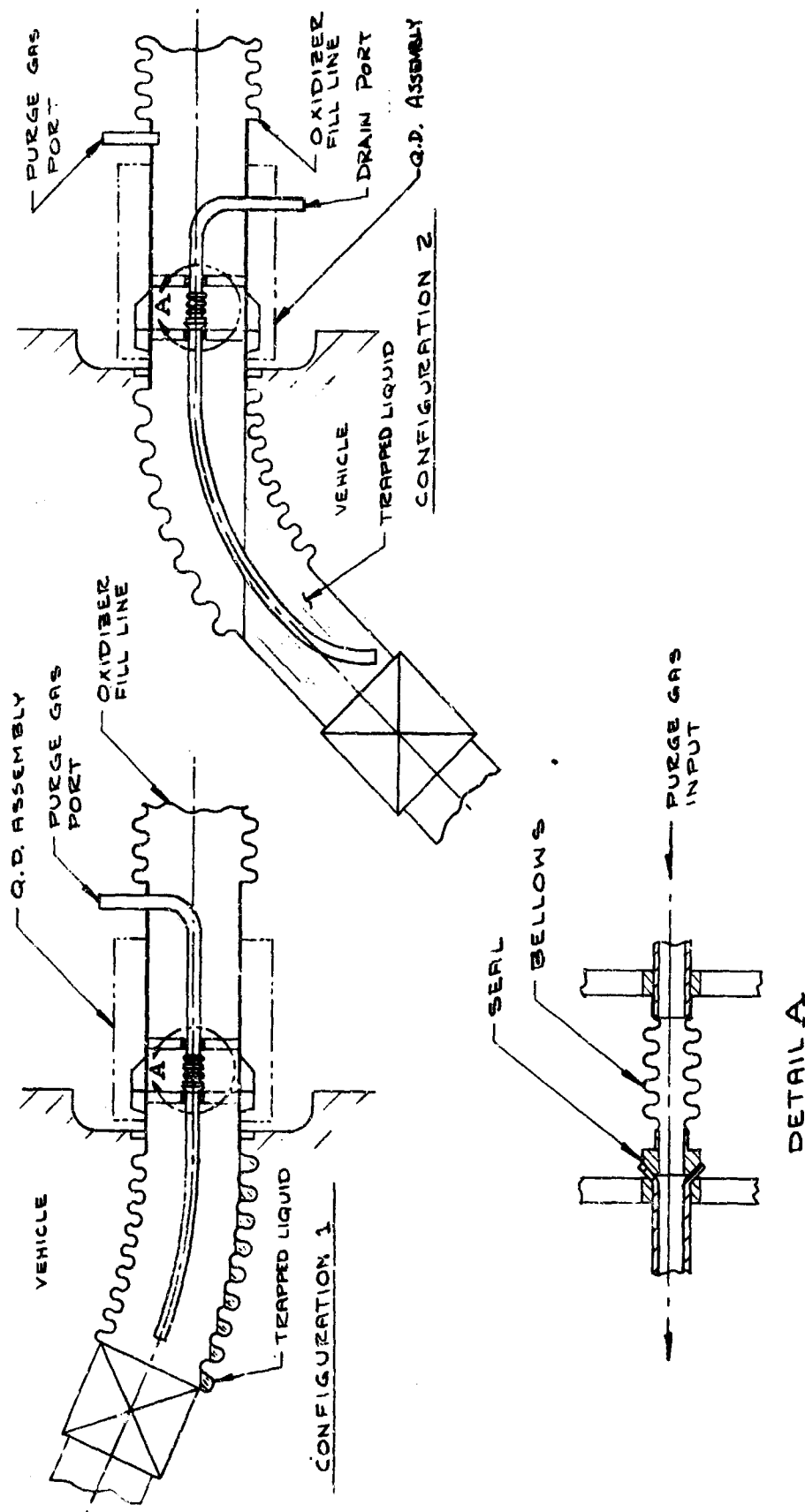


Figure 3. Vehicle Fill and Drain Line Configurations

(U) Configuration 2 results in the trapping of liquid in a low spot in the fill and drain line in addition to that trapped in the bellows. Removal of the bulk of the trapped liquid can be accomplished through use of an internal drain line positioned to drain from the low spot, as shown in Configuration 2. The liquid would be forced through this drain line by use of purge gas in the main fill line. The cycle could be reversed once the bulk of the liquid was removed, and the purge gas could be introduced through the internal purge and drain line. Implementation of either of these approaches requires the addition of a small line to the vehicle and the connection of this line to the ground (AGE) purge and drain system. Both requirements can be met easily and reliably, as shown in Detail A. By keeping this auxiliary purge and drain line and its vehicle/AGE connection inside the vehicle fill and drain line and coupling, the problem of leakage and/or spillage is eliminated, the complexity of the system minimized, and the reliability of the system is improved.

(U) All protrusions through the wall of the fill and drain line to bring in purge gases and to duct away purged fluids can be made on the AGE side of the coupling, thus minimizing vehicle system weight and complexity. The separable joint between the vehicle and AGE portions of this purge and drain line is an off-the-shelf connector used extensively by the chemical industry in making simple and effective laboratory test setups. Leakage at the connector is relatively unimportant as long as the substantial flow of the purge gas and/or purged fluids pass through the purge line. However, leakage could be minimal because the mating surfaces could be lapped to provide good sealing characteristics. Sealing loads are applied by compression of a bellows, shown in the AGE side of this line, as the fill and drain coupling is brought together and sealed. No other sealing or latching loads or connections are required with this approach.

(U) Vaporization can also be enhanced by providing a partial vacuum in the system containing the trapped liquid oxidizer. This approach is most useful where external heat sources are available to keep the liquid from becoming cooled to the point where it might freeze. In highly insulated systems, freezing is almost certain and therefore limits the effectiveness of this approach for oxidizer removal from traps in the system.

(U) Drain lines or fittings externally installed at trap points in the oxidizer system offer a positive approach to the removal of trapped liquid, even from highly insulated areas. Through appropriate valving and ducting, it is conceivable that all trap points in a system could be remotely drained of all liquid. The obvious disadvantages are increased complexity, increased weight (of particular concern for vehicle systems), and reduced reliability.

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d. Oxidizer Shutoff and Fill and Drain Valves -- Vehicle and AGE

(C) The MANSAT System Study results indicate the desirability of placing the vehicle fill and drain as well as vent valves in the highly insulated portion of the vehicle as close to the primary fluid system as possible. In so doing, the external heat leakage to the system is minimized by containing the oxidizer within the tankage and feed line and by maintaining the valve at oxidizer temperature well insulated from external heat sources by long heat paths.

(C) The mounting of these valves adjacent to the tankage or feed system precludes the trapping of (thus isolating as unusable) liquid oxidizer. Further, if fill and drain is performed through the engine feed line or through the wall of the tank in the immediate vicinity of the engine feed line connection, location of the valving adjacent to the primary oxidizer system is extremely important because it prevents the inadvertent ingestion of a gas bubble formed in the fill line into the feed system during engine operation. The introduction of such a bubble into the liquid stream feeding the engine will degrade performance and could result in destruction of the engine.

(C) In view of this, it seems evident that vehicle fill and drain as well as vent valves should be located in the highly insulated portion of the primary oxidizer system (tankage and/or feed line). Thus, these valves will be located onboard of the outer aerodynamic surfaces of the vehicle and will, therefore be, isolated by interconnecting lines (see Figure 2) from the Vehicle/AGE disconnect couplings located at the vehicle surface.

(C) Design requirements for this vehicle fill and drain valve cannot be fully specified without better vehicle and mission definition (operating pressures, operational environment, mission operations that might require valve functioning, maximum allowable loss of oxidizer during a mission, and so forth). Particularly in consideration of the MANSAT concept, however, the following should be noted:

- (1) The Vehicle fill and drain valve is required only to prevent excessive and/or hazardous escape of oxidizer from the vehicle through the fill and drain line.
- (2) The valve is actuated (cycled open and closed) many times prior to launch (checkout, testing, filling, and so forth).
- (3) The valve is not actuated following vehicle launch.
- (4) The valve does not serve as the contamination barrier between the vehicle oxidizer system and the atmosphere during ground, nonlaunch, operations (shipping, testing, checkout, connection to AGE, and so forth). The barrier to this contamination is required at the disconnect coupling to preclude contamination of the interconnecting line, the valve, and the Vehicle/AGE interface sealing surfaces.

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(5) Electromechanical valve position indication (open or closed) will be available for use to ensure that the valve has closed prior to launch.

(6) Oxidizer detectors (an appropriate unit has yet to be developed; see Section II, 10 for further details) located in the coupling and fill-line purge-gas vent would be used to detect excessive valve leakage. This greatly decreases the probability that the valve could degrade mission capabilities or cause mission failure.

(U) These considerations suggest that great confidence can be placed in knowing that the vehicle fill and drain valve has functioned properly in all ground operations, including launch of the vehicle. This implies that adequate sealing techniques have been used in the valve seat design, which is, regardless of where the valve is located, a necessary and basic requirement for all valves used in the vehicle.

(U) Solution of the other mandatory oxidizer system valve use problems (engine valves, pre-valves, vent valves, and so forth) will provide the needed technology and, potentially, even hardware for the fill and drain valve.

(U) The following is pertinent to definition of the valve on the AGE side of the disconnect coupling, and the valve's influence on the coupling:

(1) The valve must prevent the excessive and/or hazardous escape of oxidizer from the AGE portion of the vehicle fill and drain line.

(2) The valve probably will serve as the primary oxidizer flow-control device during fill, topping just prior to vehicle launch, and during draining of oxidizer from the vehicle.

(3) The valve cannot serve as the contamination barrier between the AGE oxidizer system and the atmosphere during ground, nonlaunch operations. The barrier to this contamination is required at the disconnect coupling to preclude contamination of the valve, the vehicle/AGE interface sealing surfaces, and the interconnecting line.

(4) Electromechanical valve position indication will be available for use to ensure that the valve has closed prior to launch.

(U) There do not appear to be any particular restrictions on size, weight, or location of the valve. The umbilical mast and oxidizer fill and drain line support and retraction boom can be made structurally adequate to permit location of the valve at any desired point. Location close to the vehicle/AGE disconnect coupling minimizes the amount of trapped oxidizer to be drained and purged from the line between the valve and the vehicle oxidizer fill and drain valve. This operation is discussed in Sections II, 2 and II, 11.

(U) Packaging the AGE portion of the disconnect and the AGE valve into a single unit may have several advantages (fewer external leak paths, reduced weight, and so forth). More important, however, is that the sealing and other operational characteristics should in no way be reduced or impaired.

e. Identification of Disconnect Coupling Loads

(U) The QD coupling must be designed to withstand both internally and externally applied loads that act to separate the coupling halves and/or to separate the coupling from its mounting on the vehicle. Loads from the following sources must be considered in the design:

- (1) Load Caused by Internal Fluid Pressure -- The internal pressure produces a separation load at the seal interface that must be resisted by the connecting mechanism (bolts, threads, latches, and so forth). Properly designed, the interface seal can be made to take advantage of the internal pressure to increase the sealing forces as the pressure increases. This is done by making the seal self-energizing, possibly by using a V-shape with the apex of the V oriented outward. Another method of increasing the sealing forces with pressure is to use bellows behind one of the seal seating surfaces with a larger mean diameter than the effective diameter of the seal.

An increase in the sealing force adds directly to the load on the latches. The pressure and separation load on the disconnect coupling may be amplified if a vehicle shutoff valve is used to control the propellant flow. The magnitude of pressure amplification depends on the closing rate of the shutoff valve. The location of the shutoff valve and the magnitude of the pressure amplification factor, if applicable, must be determined before final design of the disconnect coupling.

- (2) Wind Load on the Umbilical Line -- The most general method for connecting an AGE propellant servicing line to an upper stage vehicle is to use a flexible or semiflexible umbilical line for the span from the servicing mast or tower to the fill port on the vehicle. This span, of course, varies for different vehicles. When the distance between the vehicle and the mast is great, a support boom is used to provide intermediate supports for the line. The QD must be designed to support its share of the load from the final span of umbilical line and all hardware attached to the QD (actuators, valves, sensors, and so forth). A wind loading normal to the umbilical line produces a tension load, a lateral load, and a bending moment on the coupling. For cryogenic propellants, the umbilical line may be insulated with a vacuum jacket or a layer of insulation material that increases its profile area and, therefore, also increases the wind load on the line. An estimate of the wind load can be obtained by using the formula for aerodynamic drag on a cylinder in a flow field.

$$D = C_D A q$$

where

D is the drag force (pounds),

C_D is the coefficient of drag (dimensionless),

A is the profile area of the umbilical line (square feet)

and

q is dynamic pressure (pounds per square foot).

For a smooth cylinder in a low Mach number air stream, C_D is on the order of 1.2. A realistic maximum limit of wind velocity to be used for design of the umbilical is 70 mph. The drag corresponding to a 70 mph wind velocity and $C_D = 1.2$ is 15 psf. Douglas currently uses a value of 30 psf for the ultimate wind load on cylindrical umbilicals. This value provides an extra margin of safety to account for some undefinable conditions, such as whipping of the lines as a result of unsteady wind conditions and increased drag from local protuberances on the umbilical line. For analysis of loads into the coupling, it may be assumed that the flexible line has a catenary shape as a result of the uniformly distributed wind loading.

- (3) **Load Resulting from Weight of Umbilical Full of Propellant --** This is a uniformly distributed load along the umbilical line that causes the line to assume a catenary shape, as noted, for the wind loading. The direction of this load is perpendicular to the direction of the wind loading; however, the two loadings can be combined vectorially and analyzed as a single loading. A convenient method for calculating the tension loads in a catenary shape is presented in tabular form in Reference 1.
- (4) **Dynamic Load Resulting from Curvature of the Flow Path Through the Umbilical Line --** Flow through the umbilical may or may not produce a significant load on the disconnect coupling, depending on the amount of curvature in the umbilical and the velocity of flow. The magnitude of this load may be determined by use of the impulse-momentum principle of fluid mechanics.
- (5) **Load Resulting from Oscillatory Motion Between Vehicle and Umbilical Mast or Tower --** Wind loading on the vehicle and on the umbilical mast or tower produces an oscillatory motion in both that causes the distance between the support points for the two ends of the umbilical line to vary as a function of time. When the support points move apart, the catenary shape of the umbilical becomes a flatter curve and the tension in the line increases. The design analysis of the umbilical loads must

account for this maximum tension condition. The umbilical line must have sufficient length so that it is never stretched taut as a result of the oscillatory movements of the end points.

- (6) Load Required to Disconnect Coupling -- The load imparted to the coupling during decoupling depends on the design of the disconnect mechanism. If the coupling is designed to be ruptured by a tension force, this will be the maximum force experienced by the coupling. However, if a latching mechanism is used, the decoupling loads may be small compared to the other loads previously noted.
- (7) Loads Induced by Thermal Changes Within the Coupling -- A thermal gradient caused by flowing a cold fluid through the coupling generally results in contraction of the compressive members of the coupling. This causes a load decrease in the tension members of the connector, and as a result sealing loads are often accordingly reduced. The sealing load relaxation must not be so great as to result in unacceptable leakage. A detailed analytical approach to this problem is presented in Reference 2.

f. Connect Disconnect Requirements

(U) The previous portions of this section have reviewed the pertinent portions of the MANSAT Study of Reference 3, discussed vehicle/AGE oxidizer valving associated with, and imposing requirements on, the disconnect coupling, and discussed draining and purging of trapped oxidizer from the system adjacent to the disconnect coupling prior to coupling separation. The following discussion focuses on the disconnect element. Most specifically of concern are the considerations of remote as opposed to manual connection and prelaunch as opposed to fly-away disconnect.

(U) As far as could be ascertained, none of the present vehicle systems (military and space) has remotely connected vehicle/AGE interface couplings. This is true for fuel, hydraulic, electrical, pneumatic, and air conditioning disconnects, as well as those for oxidizer systems. The primary reason appears to be that no real requirement exists for remote initial connection or subsequent reconnection that justifies the complexity and attendant decrease in component reliability. Once mated and checked out, the couplings are either not disconnected until the launch is aborted or until vehicle liftoff (fly-away disconnect). If the coupling is disconnected prior to launch, abort of the launch does not impose any new hazardous condition as a result of the disconnect having been previously separated.

(U) Disconnect prior to launch of fill lines to nontoxic and otherwise safely ventable to the atmosphere cryogenic oxidizers and fluids represents no new hazard to an abort situation. However, such a

disconnection without the possibility for reconnect could be the final contributor to launch abort. Cryogenic fluids must be topped off to the 100% level and prepressurized immediately prior to launch. The time between effecting this condition and liftoff of the vehicle is carefully controlled and limited (usually a matter of a few minutes). Thus, any launch delays following propellant prepressurization usually result in venting of the propellant tank and repeating of the last portion of the countdown, including propellant topping and prepressurization. Because initiation of these recycling operations can often be effected as late in the countdown as a fraction of a second prior to engine ignition, it is impractical to separate prior to launch. To do so would impose many additional operational delays, unduly adding to the complexity and uncertainty (connection, leakage, contamination, and so forth) of the system.

(U) Coupling and system contamination is potentially the most important consideration of all. The reaction of fluorine with moisture introduces one or more of several hazardous conditions: the formation of hydrofluoric acid, an extremely corrosive substance; the formation of impact-sensitive frost-fluorine or ice-fluorine compounds; and the introduction of solid particles that could clog or otherwise functionally impair the operation and safety of the system. Therefore moisture must be prevented from forming on the fluorine wetted surfaces of the coupling. Dry inert gas shrouds, special environmentally controlled work enclosures, and so on, can be used as required to effect a moisture-free initial connection of the coupling.

(U) Remote connection with the coupling at cryogenic temperature, at which frost and ice will form rapidly from all but absolutely moisture free gases, presents many problems. The probable solutions are likely to be complex and cumbersome (large gas shrouds and enclosures), all of which result in reducing reliability, while adding to the functional and operational complexity of the system. Also, airborne contaminants can be trapped between the mating halves of the coupling much more easily during a remote connection than during an environmentally controlled manual engagement.

3. LATCHING SCHEMES

a. Latch Requirements

(U) Possible configurations for the latch and quick release mechanism for the QD coupling have been investigated. Consideration has been given to means for ensuring the attainment of the desired preload on the interface seal. One method is to use the latch as the mechanism for applying the seal preload. An alternate approach is to make the engagement of the latch the initial step in the procedure and to use an additional mechanism, such as a set of adjustment screws, to apply the desired preload to the seal. It appears that the latter approach is the more effective method because it can

accommodate a wider range of tolerances on detail parts and also permits a simpler latch design

(U) One of the more restrictive design goals is to provide a redundant release mechanism. This could be provided by incorporating a breakaway section that would rupture should the normal release mechanism fail. Another and perhaps more desirable method is to provide two independent release mechanisms at the same separation interface.

(U) The possibility of providing a safeguard to prevent inadvertent actuation of the coupling release mechanism was considered. Separation of the coupling could be disastrous if it occurred during oxidizer transfer and a significant quantity of oxidizer is spilled. Coupling separation cannot be considered hazardous if it occurs when the transfer line is dry, however, it could result in a delay of launch if it occurred in the final stages of prelaunch activities.

(U) Precautions must be taken to preclude inadvertent separation of the coupling during the oxidizer transfer operation. System design criteria should provide for an interlock between the oxidizer transfer system control and the QD release mechanism such that the QD cannot be released manually during an oxidizer transfer.

(U) Before establishing a requirement for a safety device to prevent inadvertent manual decoupling when the transfer line is dry and personnel are working in the area, the effects of such a device on the reliability of the coupling release function must be thoroughly evaluated. If such a safety device adds mechanical or system complexity, it will almost certainly lower the reliability of the coupling separation system for a normal release.

(U) On some of the latch designs considered, a shear pin could be incorporated in the latch, thus increasing the force required for unlatching and decreasing the probability of an inadvertent decoupling. One of the hazards of this method is that a shear pin of improper strength might be unintentionally substituted and prevent actuation of the unlatching mechanism. Also, the actuation forces for this type of device are directly additive to the forces required for the normal unlatching. Because the normal unlatching force is a highly variable quantity as a result of its dependence on frictional effects and possible galling of sliding surfaces, the total force required for unlatching might become excessively great.

(U) Another scheme for a safety device to reduce the possibility of inadvertent manual decoupling is to design a breakaway device (such as a shear pin) that is broken before the unlatching load is transmitted to the latch. This could be accomplished by a slack cable or a slotted linkage that breaks the safety device during its initial motion.

(U) Although the inadvertent decoupling of a dry line does not result in a hazardous situation, it could stop or delay a launch, and either a safety device should be provided or the latch design should be such that inadvertent manual actuation is virtually impossible.

(U) Another safety requirement for the latching mechanism is an electrical feedback signal to the blockhouse indicating that the QD is properly latched and ready for propellant transfer. This easily could be provided on any of the latching mechanisms considered by use of limit switches to indicate that the critical components of the QD are in the connected and locked position. Strain gages could be used to ensure that seal loads have been effected.

(U) All of the latch designs considered will be vulnerable to possible jamming and higher unlatch loads if ice is allowed to form on the cold surfaces as a result of precipitation and freezing of atmospheric water vapor. Ice or frost also represents an additional hazard because it will become impact sensitive if any fluorine leaks from the transfer line is absorbed by it. For these reasons, it will be necessary to exclude the atmospheric air from the vicinity of QD coupling during transfer of the cryogenic oxidizers LF_2 and FLOX. It appears that the most feasible method for excluding air is to provide a shroud around the QD coupling and purge it with a dry inert gas, such as N_2 .

(U) To minimize the potential fire hazard that could be created if even a minor amount of F_2 escapes from the transfer system, only F_2 -compatible materials should be used in the construction of a shroud. The use of Teflon in limited quantities is permissible if seals are needed to reduce gross leakage. A continuous flow of the relatively warm purge gas is necessary to prevent the shroud itself from becoming cold and building up a layer of ice that could cause operating problems with the latch actuating system.

(U) A sketch of a possible shroud configuration is shown in Figure 4. It consists of a cylindrical metal bellows sufficiently large to enclose the QD coupling and its latch mechanism. The bellows is supported by a bulkhead that attaches to the AGE-half of the coupling. To provide access to the latching mechanism when the coupling is being mated, the shroud bellows is moved axially along the umbilical hose to uncover the latching mechanism. After the coupling halves have been mated and the interface seal preloaded and leak checked, the shroud is moved to its normal position with the bellows lightly compressed against the vehicle and clamped to its supporting bulkhead.

b. Evaluation of Latch Designs

(U) An evaluation of five configurations for the QD coupling latch and quick release mechanism is presented in the following discussion. A comparison of the merits of each of these configurations is presented in Table I. The same basic interface seal has been

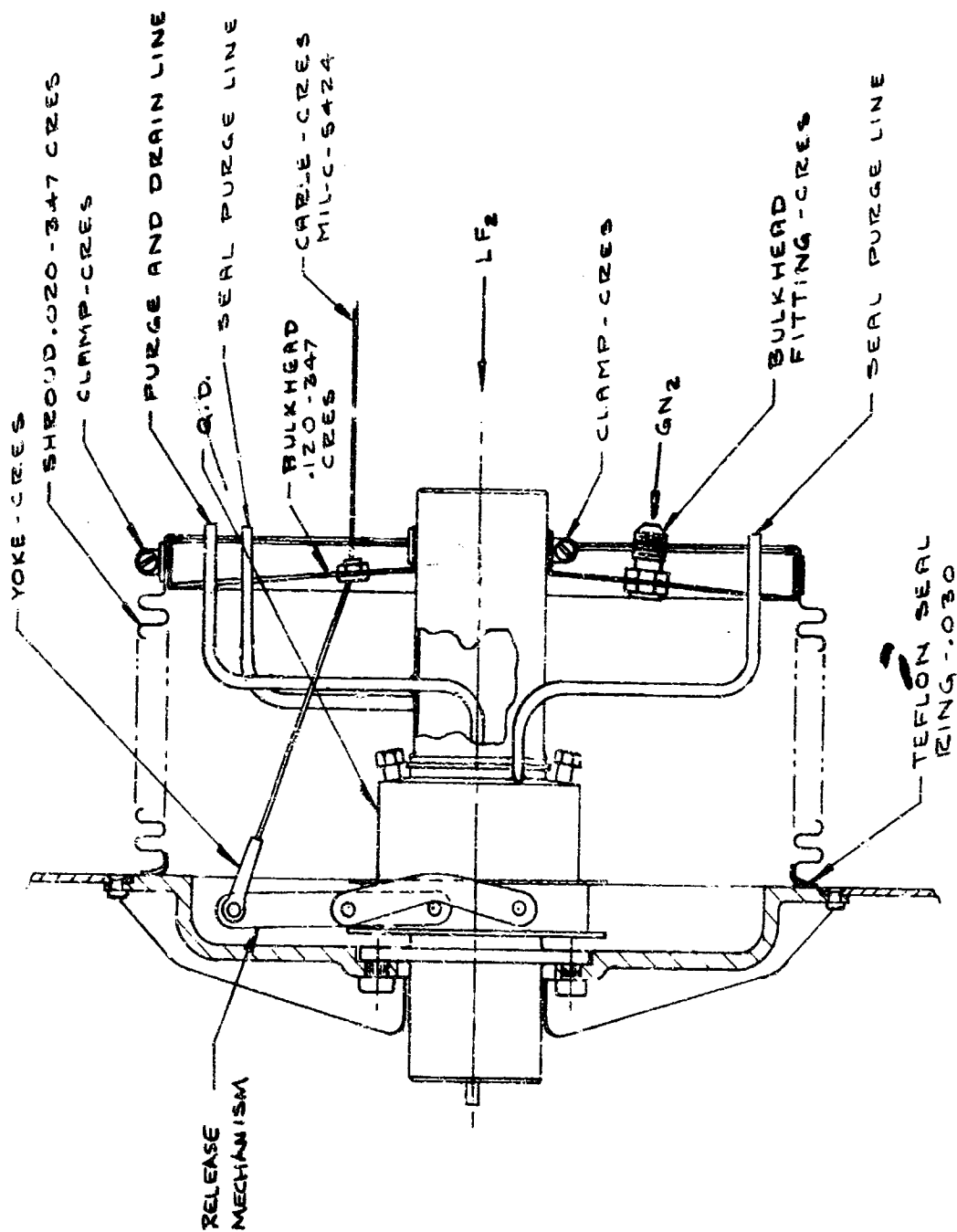


Figure 4. General Arrangement of Shrouded Test QD Coupling

Table I
COMPARISON OF MERITS OF LATCH CONFIGURATIONS (U)

Configuration No.	Description	Unlatching Load (Total)		*Advantages	Disadvantages
		Unlubricated	Lubricated		
I	Ball latch with expanding retainer band	1116#	171#	1. Provides redundant release feature since actuation of any one of the three latches will permit coupling to separate. 2. Precision balls are commercially available at relatively low cost. 3. Precision balls are commercially available at relatively low cost.	1. High contact stresses generated by the balls are usually accompanied by brinelling or galling of the mating surfaces unless they can be made of a very hard material. 2. Dependent on low friction coefficients for reasonably low release load. 3. Release loads are relatively high with or without lubrication on sliding surfaces. 4. The coupling will not release if the retainer sleeve "hangs up."
II	Ball latch with sliding retainer sleeve	5364#	711#	1. Motion of the retainer sleeve during the release phase positively opens the latching fingers and permits unobstructed release of the vehicle-half flange. 2. Adequate finger bearing area to prevent brinelling of the vehicle-half flange can be provided easily.	1. Not readily adaptable to incorporation of a redundant release scheme. 2. High contact stresses generated by the balls are usually accompanied by brinelling or galling of the mating surfaces unless they can be made of a very hard material.
III	Finger latch with sliding retainer sleeve	1272# + spring load	372# + spring load	1. Provides redundant release feature since actuation of either of the two latches will permit coupling to separate. 2. Not dependent on low friction coefficients for low release loads. 3. Adequate clamp bearing area to prevent brinelling of the vehicle-half flange can be provided easily.	1. Same as for configuration II. 2. Same as for configuration II. 3. Same as for configuration II.
IV	V-band clamp with over-center toggle latches	206#	79#	1. Motion of the release ring during the release phase positively opens the latching fingers and permits unobstructed release of the vehicle-half flange. 2. Adequate clamp bearing area to prevent brinelling of the vehicle-half flange can be provided easily.	1. Accurate seal preload values will be difficult to determine because they are dependent on a highly variable parameter, the frictional resistance between the outer clamp ring (C) and the adjustable lugs (D).
V	Finger latch with over-center toggle lock	855#	228#	1. Not adaptable to incorporation of a redundant release scheme. 2. Dependent on low friction coefficients for reasonably low release load.	1. Not adaptable to incorporation of a redundant release scheme. 2. Dependent on low friction coefficients for reasonably low release load.
<p>*All of the latch configurations have the following desirable features:</p> <ol style="list-style-type: none"> 1. After the latch is released the coupling will not "hang up" as a result of a pitch or yaw motion along with the axial separation motion. 2. Preloading of the seal is independent of the latching operation thereby simplifying the latch requirements. <p>†Possibly the retainer sleeve could be made frangible so that a load higher than the normal operating load would cause it to rupture.</p>					

(U)

assumed for all of the configurations. The seal consists of a soft metal gasket that is plastically deformed on each side by two concentric knife-edge serrations machined into the hard metal flanges of the coupling. The soft seal is constrained within the AGE-half of the coupling by a wire retaining ring. For the purpose of evaluating the latch release loads for each of the configurations, the seal preload was calculated as 8,200 lb. This is based on a total seal length of 13.7 in. and a sealing load of 600 lb/in. Figure 20 in Section II, 5 indicates that a seal load of 200 lb/in. would provide a leakage rate of 10^{-6} SCIM of helium. However, to maintain a seal of this quality, a higher loading is required to account for stress relaxation of the seal material caused by temperature cycling and by external loads on the QD. A factor of three was assumed to assure that an adequate sealing stress would always be maintained. It is likely that this seal loading can be reduced when a specific design is more completely analyzed.

(U) The knife-edge serrations on each half of the coupling are afforded some degree of protection from physical damage by recessing the sealing surface from the flange face. The flange faces are designed to nest within each other to provide radial alignment of the two coupling halves.

(U) It has been assumed that the actuator for the latches is not integral with the QD but is mounted on the umbilical servicing mast and connected to the latches by means of a cable harness. If necessary, the latch actuator could be attached directly to the AGE-half of the QD coupling. This would greatly increase the complexity of the QD coupling and also the mass of equipment that must be retracted at time of launch. If a suitable mounting can be made on the mast, this location is undesirable.

(1) Latch Configuration I

(U) The Configuration I latching and release mechanism is shown in Figure 5. The vehicle half of the coupling consists of a simple flanged fitting (A) containing the two sealing knife edges on its interface surface. The AGE half of the coupling contains the soft metal seal (K) and the latching and release components. The latching components consist of six 1/2-in.-dia balls (G) fitted into radial holes in the body (C) of the coupling. When the two halves of the coupling are mated, the balls are forced into a circumferential groove in the vehicle half flange (A). The balls are locked into place by the band (D) and the three over-center toggle latches made up of links (E) and arms (F). Preloading of the seal is accomplished by torquing the bolts (H) to the desired preload value. To release the coupling, the arms (F) are pulled by means of cables attached to actuators. Actuation of any one of the three latches will disconnect the coupling. The retaining pins (J) restrict the travel of the band (D) so that the balls cannot escape from

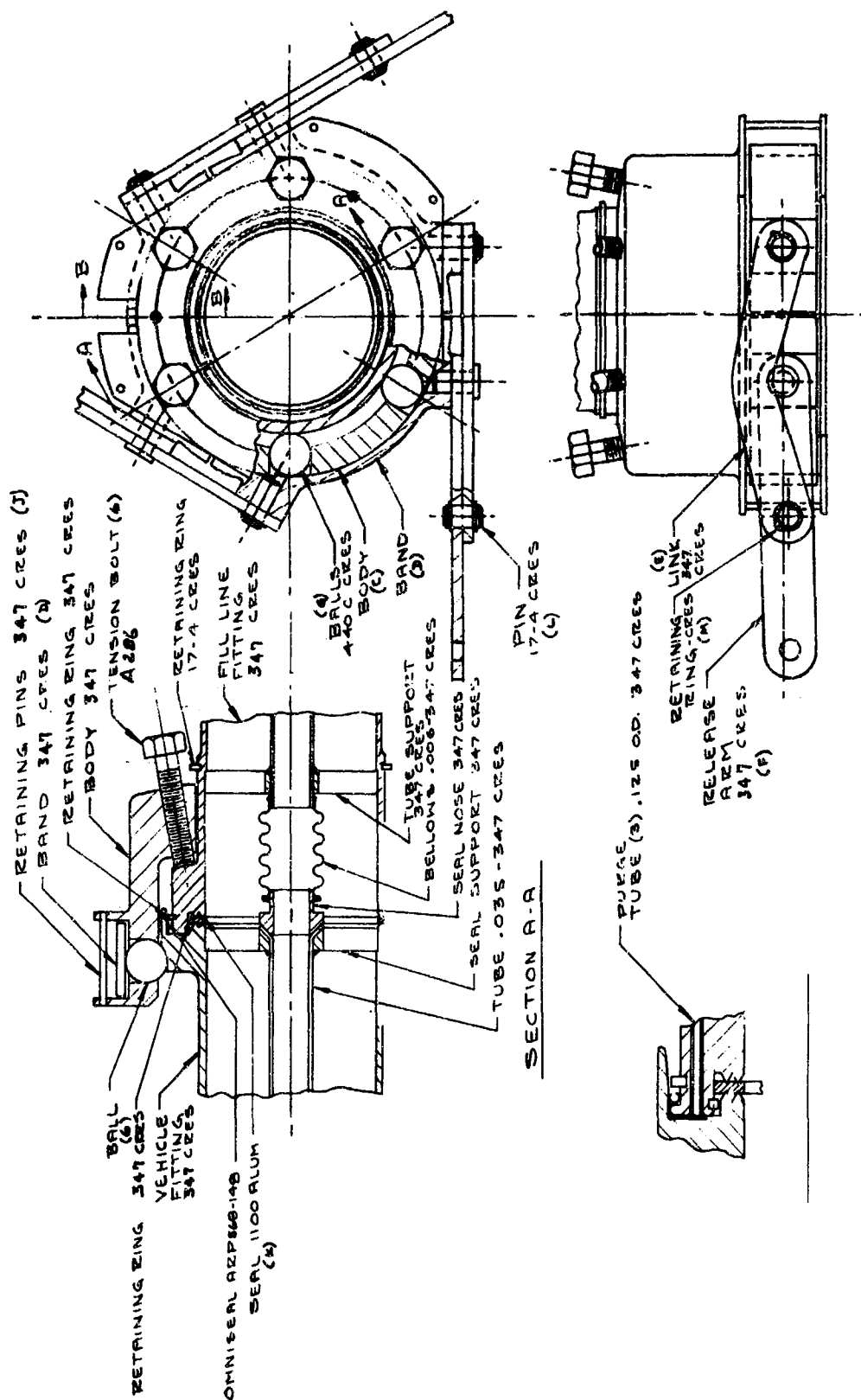


Figure 5. Latch Configuration I--Ball Latch with Expanding Retainer Band

their positioning holes when the coupling is unlatched. The maximum depth of engagement of the balls with the flange of fitting (A) must be such that frictional effects will not cause the balls to hang up when the over-center latches are released. Figure 6 shows the loads on the various components of the latching mechanism assuming (1) that no lubrication is used and (2) that all sliding surfaces are lubricated with molybdenum disulfide. Case Numbers 3 and 5 in Figure 6 are ideal, cases assuming no friction, and are included to indicate the minimum unlatching loads that might be obtained if a highly efficient lubricant such as an oil or grease could be used. For a design using no lubricant and the assumed friction coefficient of 0.78, the ball loading angle always must be 38° or larger, otherwise the balls will remain locked after the band is released.

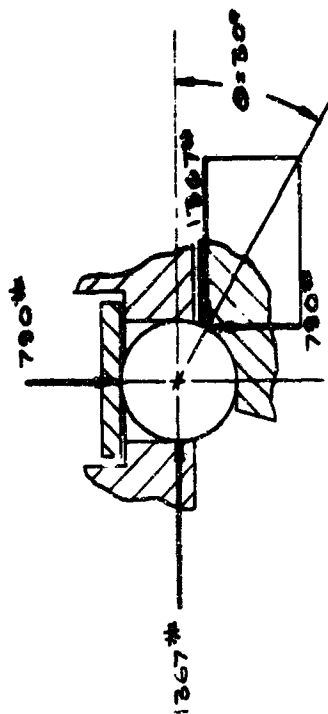
(2) Latch Configuration II

(U) The Configuration II latching and release mechanism is shown in Figure 7. The vehicle half of the coupling is essentially the same as that for Configuration I. The AGE half of the coupling contains the soft metal seal (L) and the latching and release components. The latching components consist of six 1/2-in. -diam balls (E) fitted into radial holes in the body (C) of the coupling. When the two halves of the coupling are mated, the balls are locked into the latched position by the spring loaded sleeve (D). Preloading the seal (L) is accomplished by uniformly torquing the bolts (K) in the same manner as for Configuration I. A cable harness consisting of three equally spaced cables (N) connects the ball locking sleeve (D) to an actuator. The coupling is released by pulling the sleeve axially until a circumferential groove in its inner surface is in line with the balls and provides space for them to move out of their latched position. The sleeve is stopped before the balls are completely uncovered and free to escape from their positioning holes by compressing the spring (F) to its solid height against the retainer ring (H). Continuing pull by the cables after the sleeve is fully retracted forces the balls out of their latched position and separates the two coupling halves. The same consideration as noted for Configuration I for establishing the maximum depth of engagement for the balls is applicable. Figure 8 shows load diagrams for two conditions: (1) no lubrication and (2) all sliding surfaces lubricated with molybdenum disulfide.

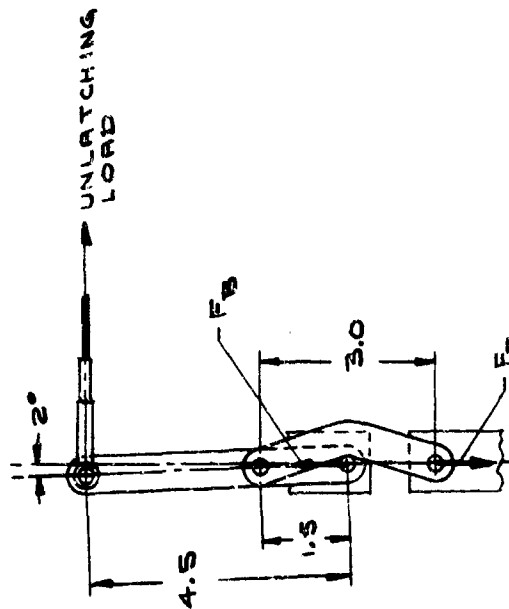
(3) Latch Configuration III

(U) The Configuration III latching and release mechanism is shown in Figure 9. The vehicle half of the coupling is similar to Configurations I and II, except that the latch engaging surface of the flange of fitting (A) is tapered rather than grooved. The latching mechanism consists of a series of metal fingers (D) that hook over the tapered flange (A) and are locked in position

(a) LOAD DIAGRAM FOR 40° LOADING
ANGLE ON LATCHING BALL



(b) LOAD DIAGRAM FOR 30° LOADING
ANGLE ON LATCHING WALL



(C) LOAD DIAGRAM FOR LATCH

CASE NO	θ	BAND LOAD FB	μ_s	UNMATCHING PRE-LOADING LOAD	TOTAL UNMATCHING LOAD
1	40°	1145#	0.78 (REV)	372#	1116#
2	40°	1145#	0.18 (NOM)	725#	218#
3	40°	1145#	0	173#	40#
4	30°	790#	0.18 (NOM)	57#	171#
5	30°	790#	0	9#	27#

*MOLYBDENUM DISULFIDE.

Figure 6. Load Diagrams for Latch Configuration I

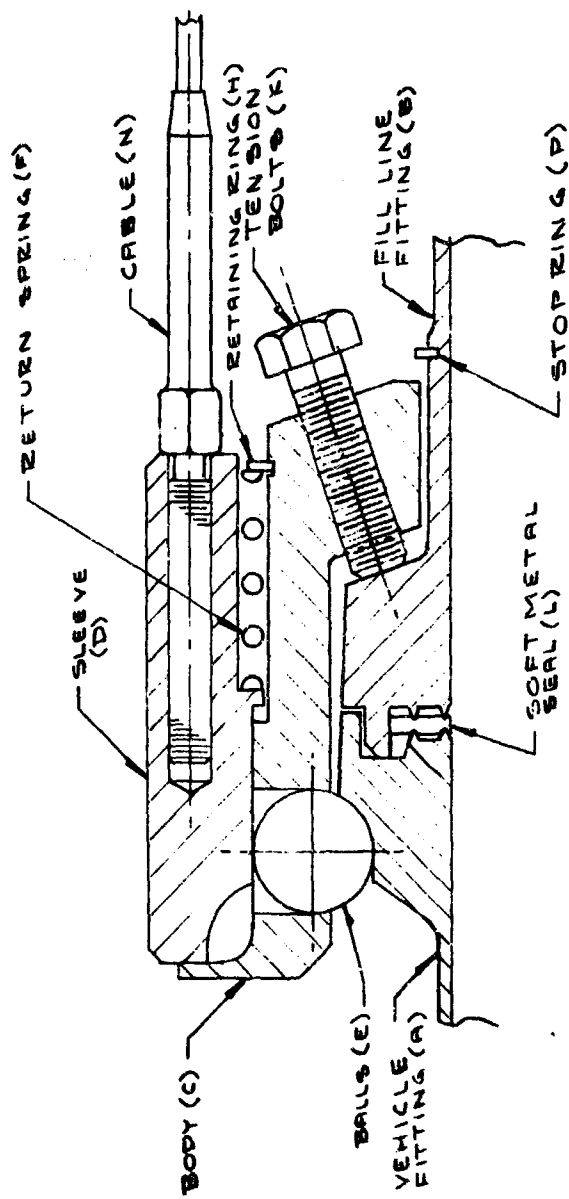


Figure 7. Latch Configuration II--Ball Latch with Sliding Retainer Sleeve

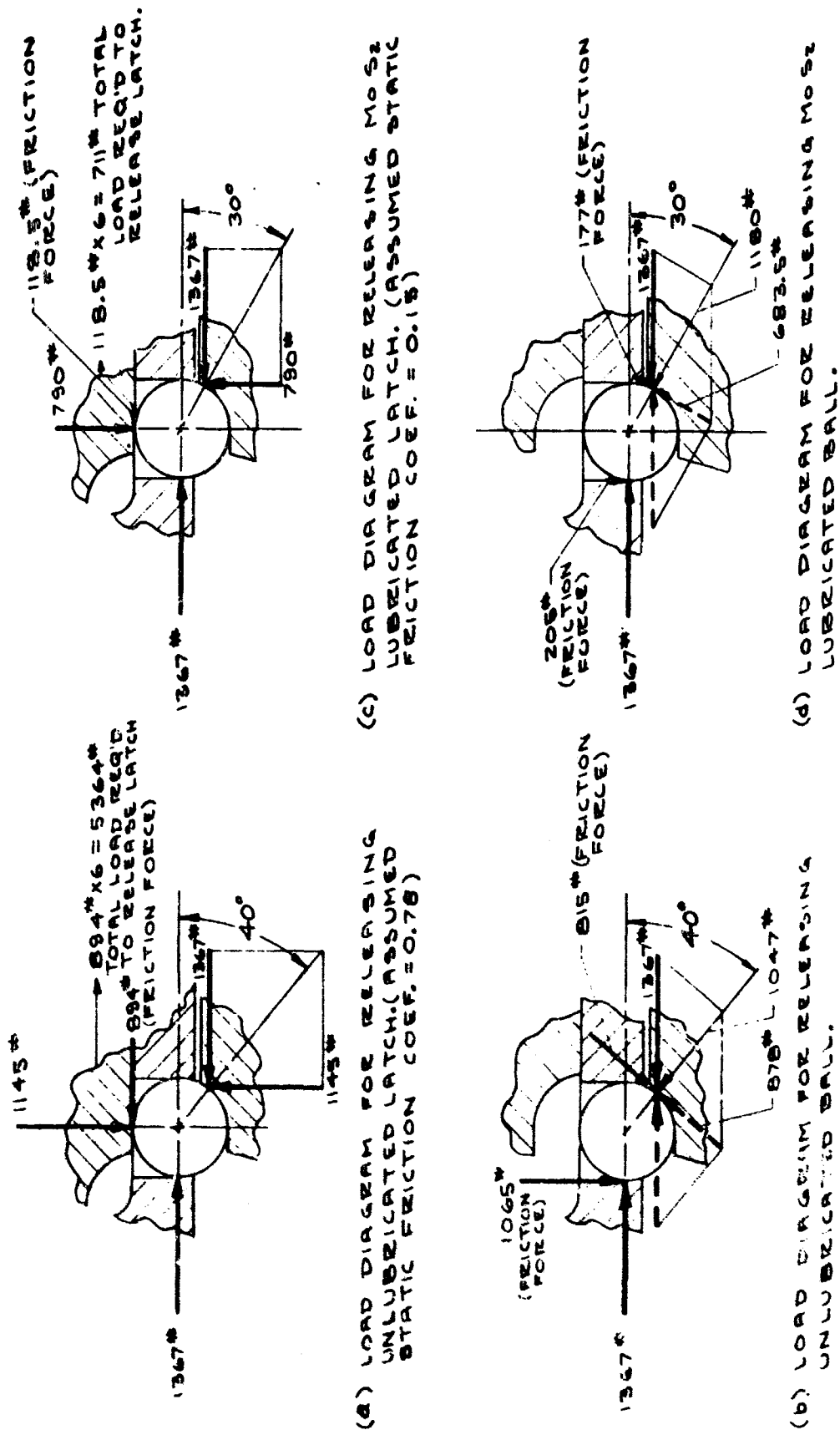


Figure 8. Load Diagrams for Latch Configuration II

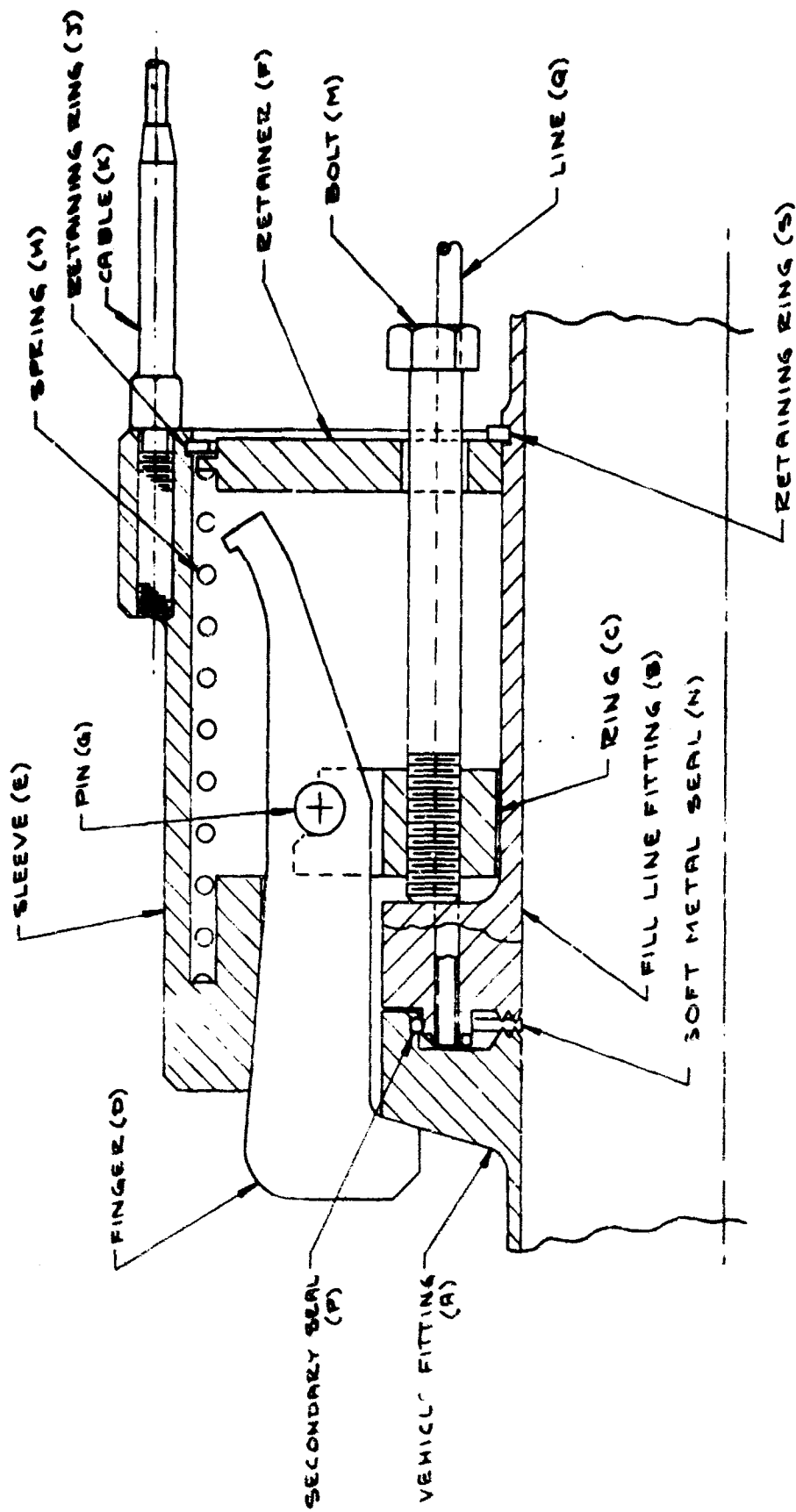


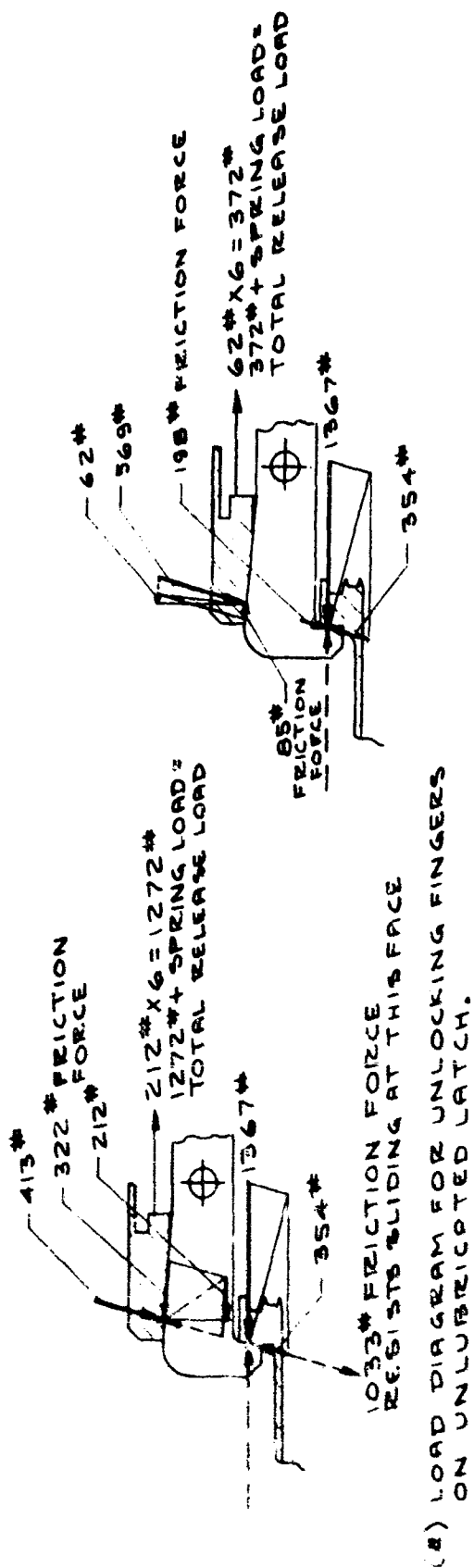
Figure 9. Latch Configuration III--Finger Latch with Sliding Retainer Sleeve

by a spring loaded sleeve (E). Preloading the seal (N) is accomplished by uniformly torquing the bolts (M) in the same manner as for Configurations I and II. A cable harness consisting of three equally spaced cables (K) connect the latch locking sleeve (E) to an actuator. Coupling release is effected by pulling the sleeve axially. Because the inner surface of the sleeve is conical, its initial motion relieves the positive loads holding the fingers in the latched position. However, frictional forces between the fingers and the fitting (A) may prevent immediate disengagement. After the sleeve (E) has moved past the pivot pins (G) it engages the ramps on the fingers (D) and provides a positive opening force to cause the fingers to release the fitting (A).

(U) Also shown on the configuration is a secondary seal (P) made of Teflon and placed outside of the primary seal. Ports are provided to the cavity between the two seals, and line (Q) provides for vacuum scavenging of any oxidizer leakage past the primary seal. This is an alternate sealing concept that could be used if the single seal concept does not prove adequate. Figure 10 shows load diagrams for two conditions: (1) no lubrication and (2) all sliding surfaces lubricated with molybdenum disulfide.

(4) Latch Configuration IV

(U) The Configuration IV latching and release mechanism is shown in Figure 11. The vehicle half of the coupling is the same as for Configuration III, except that the flange is tapered at a larger angle from the plane of the coupling interface. The latching mechanism consists of two identical halves of a V-band clamp that fits over tapered flanges on the two mating halves of the coupling. The V-band clamp ring halves are locked together with two over-center toggle latches. After the clamp is locked into place, the seal (H) is preloaded by torquing the bolts (G). The body of the clamp (C) is slotted between the bolts (G) to provide sufficient flexibility so that the clamp can expand when the latches are unlocked. The design requires that the clamp be machined to a diameter larger than its installed diameter so that when either of the latches are released it will expand to a diameter large enough to slip off of flange (A) on the vehicle and permit the coupling to separate. Lugs (D) form the movable side wall of the clamp. They are tapped to receive the seal preloading bolts (G). To release the coupling, the levers (F) are pulled by means of cables attached to actuators. This moves the toggle links (E) out of their over-center locked position and opens the clamp. The slopes of the side walls of the clamp are designed so that frictional effects will never prevent release of the clamp after either of the toggle latches have opened. Figure 12 shows load diagrams for



(c) LOAD DIAGRAM FOR UNLOCKING FINGERS ON MOD2 LUBRICATED LATCH.

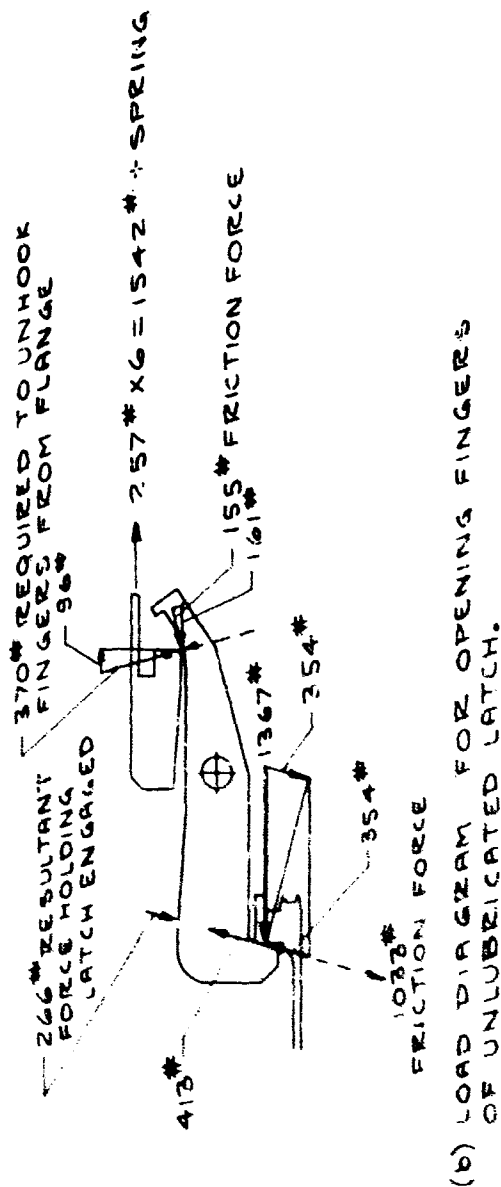


Figure 10. Load Diagrams for Latch Configurations III

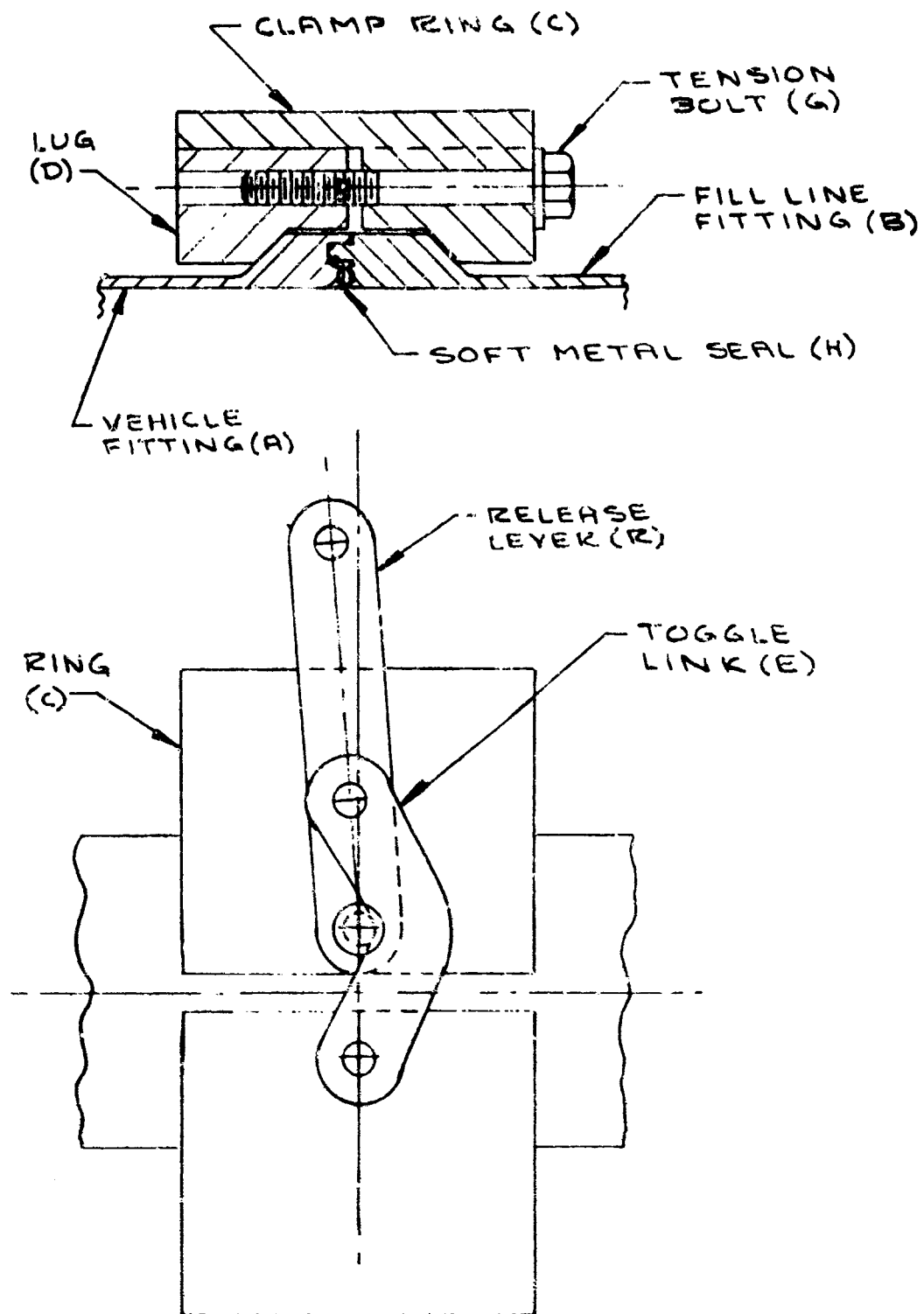
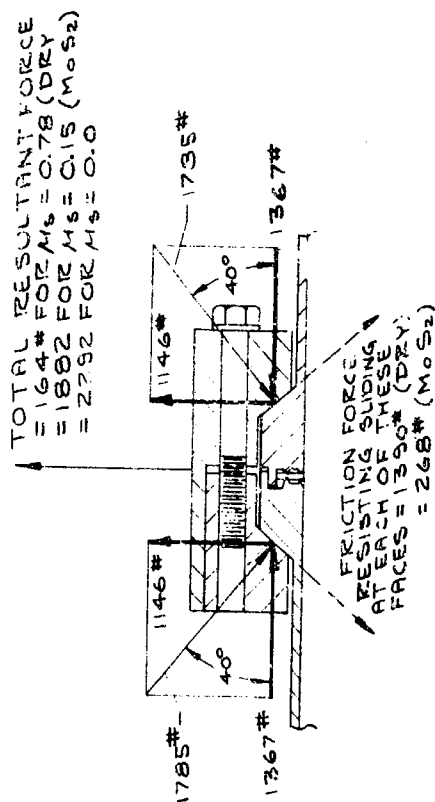
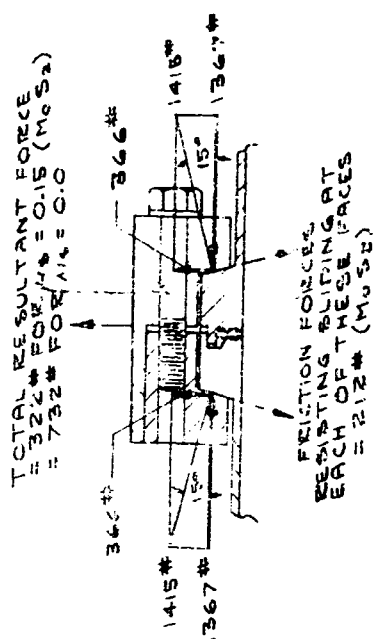


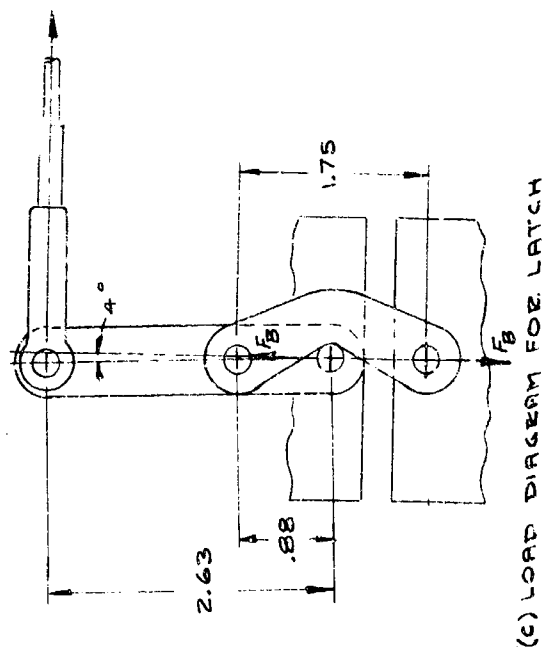
Figure 11. Latch Configuration IV V-Band Clamp with Over-Center Toggle Latches



(a) LOAD DIAGRAM FOR 40° TAPERED FLANGE



(b) LOAD DIAGRAM FOR 15° TAPERED FLANGE



EFFECT ON UNLATCHING LOAD OF VARIATIONS
IN FLANGE TAPER ANGLE (θ) AND STATIC
FRICTION COEFFICIENT (μ_s)

CASE NO.	θ	μ_s FOR FLANGE	F_B BAND LOAD	μ_s W.C.Z. LATCH	UNLATCHING LOAD PER LATCH	TOTAL UNLATCHING LOAD
1	40°	0.78 (DRY)	254	0.78 (DRY)	103#	261#
2	40°	0.78 (DRY)	275#	0.15 (μ_{OS_2})	25#	50#
3	40°	0.15 (μ_{OS_2})	1993#	0.15 (μ_{OS_2})	182#	364#
4	40°	0	2406#	0	56.5#	113#
5	15°	0.15 (μ_{OS_2})	433#	0.15 (μ_{OS_2})	39.5#	78#
6	15°	0	843#	0	20#	40#

* MOLYBDENUM DISULFIDE

Figure 12. Load Diagrams for Latch Configuration IV

two conditions: (1) no lubrication used and (2) all sliding surfaces lubricated with molybdenum disulfide.

(5) Latch Configuration V

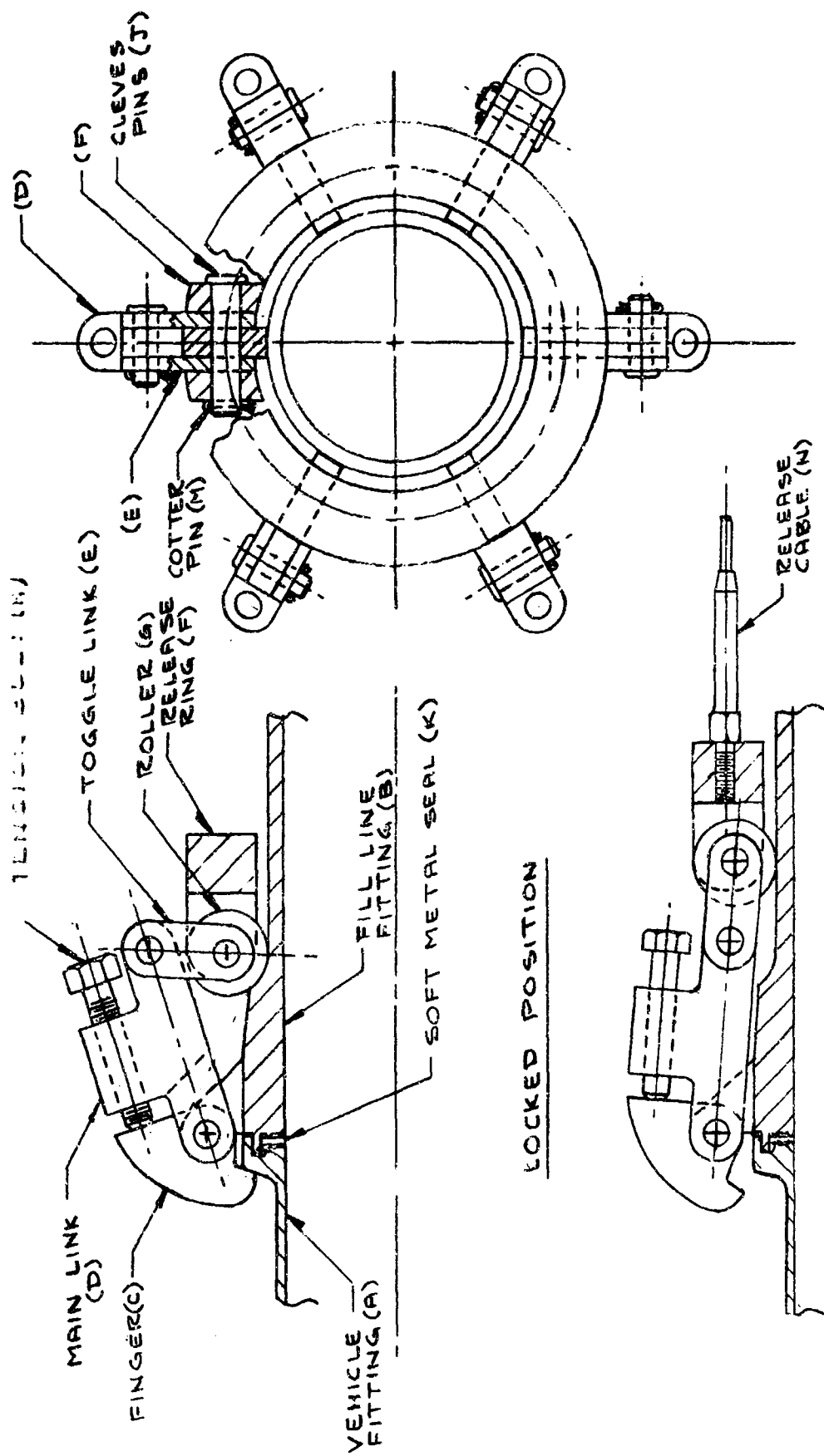
(U) The Configuration V latching and release mechanism is shown in Figure 13. The vehicle half of the coupling is similar to that for Configuration III. The latching mechanism consists of six metal fingers (C) that hook over the flange on the vehicle fitting (A). A set of toggle links (D and E) forms an over-center lock for each finger. The roller (G) is utilized as an antifriction device for the unlatching mode. The seal (K) is preloaded by uniformly torquing the bolts (N) against the fingers. The release ring (F) is attached to the inboard pivot point of each of the toggle links (E). A cable harness consisting of three equally spaced cables (N) connects the release ring to an actuator. Coupling release is effected by pulling the release ring axially, which unlocks the toggle links and permits the fingers to open and release the flange (A). The release system can be made semiredundant by attaching two independent actuation devices to the release ring. Because the release ring is not in sliding contact with any other member, it is not vulnerable to hang up.

(U) Because the latch mechanism is AGE and its weight is not critical, all of the components can be over designed so that the possibility of a rupture-type failure would be extremely remote. The only single failure that could prevent release would be a lock up of a joint in one of the finger latches or toggle linkages preventing movement of the release ring. By providing a functional checkout of the release mechanism during the preflight checkout, the possibility of this type of failure could be virtually eliminated. Figure 14 shows load diagrams for two conditions: (1) no lubrication used and (2) all sliding surfaces lubricated with molybdenum disulfide.

c. Conclusions

(U) Based on a review of the merits presented in Table I, Configuration I appears to be the best overall concept for a QD coupling for use with any of the fluorine-related oxidizers. Its chief advantage over three of the other configurations is its redundant-release feature. Although Configuration IV also has a redundant-release feature, its mechanism for preloading the seal is unattractive. The frictional effects between the clamp ring (C) and the adjustable lug (D) are inherently so highly variable that the seal preload cannot be predicted with any degree of accuracy.

(U) All of the concepts except Configuration IV require use of a lubricant on their sliding surfaces to obtain a reasonable low release load. This is a direct result of the high seal-compression preload (8,200 lb) calculated as a requirement for the type of seal chosen. With additional analytical work it may be possible to reduce this



OPEN POSITION

Figure 13. Configuration V-- Finger Latch with Over-Center Toggle Lock

loading requirement. If a suitable seal requiring less preload could be used, the latching loads would be reduced in direct proportion to the reduction in preload. For instance, if a single rather than a double knife-edge seal is used, the required preload would be reduced by half. Additional effort should be expended in Phase II to either reduce the preload or to improve the release concept. Because the assumed dry lubricant molybdenum disulfide (MoS_2) is not compatible with fluorine, it should not be used if a suitable alternate solution can be found.

(U) Configuration V is attractive because it can be packaged compactly, could be expected to give consistent results on repeated operations, appears to have a high probability of disconnecting normally, and has a reasonably low release load with MoS_2 . Configurations II and III are the least attractive because of their relatively high release loads, because they appear to be the most vulnerable to seizure as a result of metal galling, and because they do not have a redundant-release feature.

4. SEALING REQUIREMENTS

(U) The design criteria of a quick disconnect assembly suitable for fluorine service usually includes a no-leak capability. Experience with the best designed QD assemblies developed for transfer of cryogenic liquids (or corrosive cryogenics) indicates that complete sealing to achieve repeated no-leak performance is virtually impossible.

(U) A more realistic approach must accommodate the view that leaks are going to occur as a consequence of (1) limitations on seal efficiency during pressure and temperature cycling, (2) corrosive action of the oxidizer on sealing surfaces (reactivity, effects of trace impurities, and so on), and (3) unanticipated mechanical malfunctions.

(U) The allowable leak rate, which should be as low as possible without prejudicing reliability, probably depends on considerations of the toxic hazards and hypergolicity problems arising from the intermixing of small amounts of oxidizer with air or fuel.

(U) Little data are currently available to assess the true nature of the hypergolicity problem, but certain engineering design steps can be instituted to prevent air mixing of oxidizer and fuel in the vicinity of the QD device, the service tower, and within the vehicle. These factors are discussed further in Section III.

(U) When minor malfunctions or system anomalies occur, leak criteria are of importance since personnel may have to go into the area to correct the situation to continue a countdown or return the system to a standby condition.

(U) If the toxic hazard is, indeed, the principal factor that must be considered, a limiting value for the leak rate must be determined to define the engineering design requirements. An approach to defining an allowable leak rate follows.

(U) Adopting a worst case rationale, let the following conditions be assumed at the QD:

- a. Absolutely stagnant (isotropic) atmospheric conditions: no temperature convection or wind or air movement.
- b. Leak rates of fluorine (for example) ranging from 10^{-4} to 10^{-6} scim.
- c. The leak is constant and originates from a single orifice or point.
- d. Standard conditions of temperature and pressure exist.
- e. Maximum allowable concentrations of fluorine from 1 to 3 ppm are in the vicinity.

(U) Figure 15 illustrates the geometrical model for evaluating the rate of diffusion and mixing of fluorine with air and the rate of concentrational advance radially outward with time.

$$\nabla^2 \phi - \frac{1}{D} \frac{\partial \phi}{\partial t} + C = 0$$

where

ϕ is any function dependent on time (t) and distance (r),
D is the diffusion constant, and
C is a constant.

One solution of the above differential equation is:

$$\phi = \frac{1}{\sqrt{t^3}} e^{-\frac{r^2}{4Dt}}$$

where

r is the distance.

Partially differentiating ϕ with respect to time gives

$$\frac{\partial \phi}{\partial t} = \frac{e^{-\frac{r^2}{4Dt}}}{2t^{\frac{5}{2}}} - 3 \left(\frac{r^2}{2Dt} \right)$$

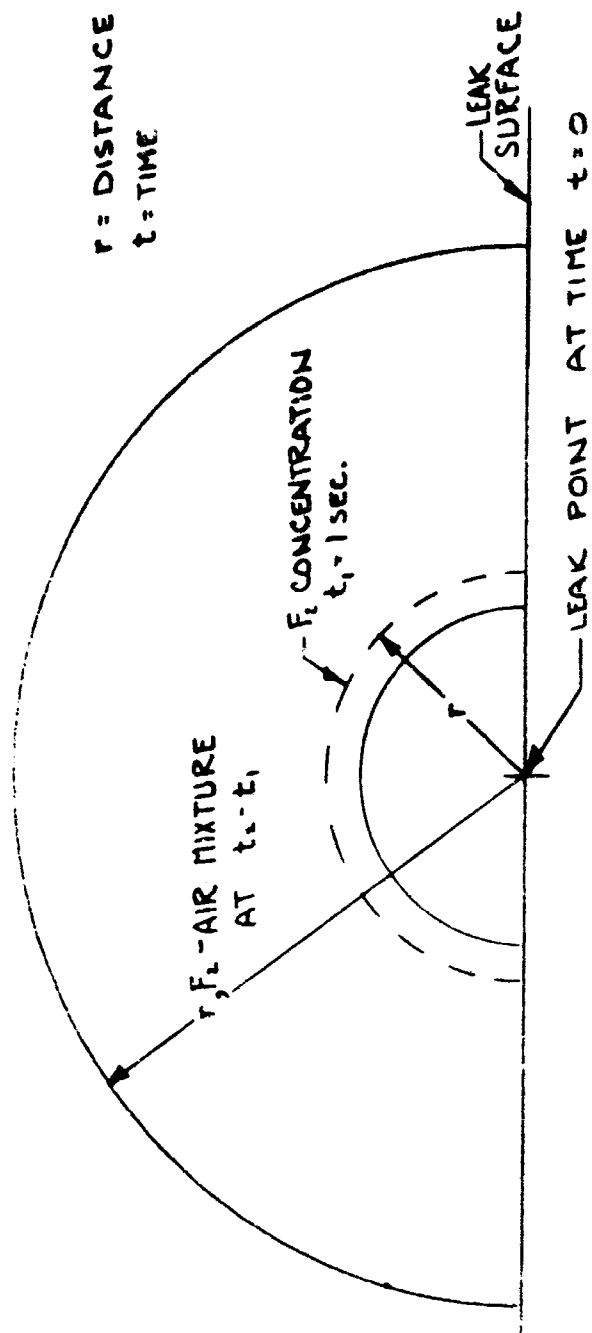


Figure 15. Diffusion Model for F_2 Leak

Now, ϕ is defined as the concentration.

At the time of maximum concentration

$$\frac{\partial \phi}{\partial t} = 0$$

and

$$\left(\frac{r^2}{2Dt} \right) - 3 = 0$$

or

$$t = \frac{r^2}{6D}$$

D, the diffusion coefficient, was estimated by the method of Othmer and Chen (Reference 4) using the critical molar volumes of fluorine and the average for air, giving 0.13 sq cm/sec.

(U) At arms length, $r \approx 60$ cm, substituting for r and 0.13 sq cm/sec for D in the last expression:

$$t = \frac{r^2}{6D} = \frac{3,600}{0.78} = 4,620 \text{ sec}$$
$$= 1.28 \text{ hours}$$

(U) After the first sec, $t + 1$ sec, at 10^{-2} cc/sec leak rate, 0.01 cc occupies a volume with a radius $= 1.33 \times 10^{-1}$ cm.

For steady state leak conditions, the flow through each advancing shell is constant and concentration, ϕ , falls off as r^{-2} .

(U) Assuming that pure F_2 is present in the volume with $= 1.33 \times 10^{-1}$ cm at $t + 1$ sec arms length (60 cm)

$$\phi \frac{F_2}{\text{Air}} = \left(\frac{1.33 \times 10^{-1}}{60} \right)^2 = \frac{1}{2 \times 10^5} \quad (2)$$
$$= 5 \times 10^{-6} = 5 \text{ ppm (V/V)}$$

occurring in 1.28 hours.

The formula provides a unique solution at each fixed constant leak rate in terms of concentration, distance, and time.

(U) Table II lists the values obtained by computer solution of this equation.

(U) Under the assumed conditions, evaluation of the toxic hazard for close emergency work requires comparison of the acceptable Emergency Exposure Limits (EEL's) with the values of concentration, distance, and elapsed leak time calculated from the diffusion model equation.

Table II

F₂ CONCENTRATION AS A FUNCTION OF DISTANCE WITH TIME OF
DIFFUSION MIXING WITH AIR (U)
(Based on Diffusion Model-Isotropic Conditions)

Concentration (ppm F ₂)	Leak Rate (SCIM)	Distance (Inches)	Time (Hours)
1.0	10 ⁻²	133.65	41.04
	10 ⁻⁴	28.79	1.90
	10 ⁻⁵	13.36	0.41
	10 ⁻⁶	6.20	0.08
2.0	10 ⁻²	94.50	20.52
	10 ⁻⁴	20.36	0.95
	10 ⁻⁵	9.45	0.20
	10 ⁻⁶	4.38	0.04
3.0	10 ⁻²	77.16	13.68
	10 ⁻⁴	16.62	0.63
	10 ⁻⁵	7.71	0.13
	10 ⁻⁶	3.58	0.02

(U)

(U) Recalling the EEL's recommended for fluorine by NAS-NRC (3 ppm for 10 min, 2 ppm for 30 min, and 1 ppm for 60 min), a more or less objective comparison can be made. Thus, it appears that total elapsed leak times ranging from 0.6 hours to approximately 8 hours would not result in concentrations much above 1 to 3 ppm at approximately normal working distance (30 cm), when the leak rates are in the range of 10^{-4} or 10^{-5} scim.

(U) The worst-case isotropic conditions used to develop the argument for selection of a reasonable leak criteria would almost never occur in practice. Range safety would not permit operations to proceed without a minimum wind velocity and the proper wind shift and vertical temperature-lapse conditions predicted for assuring maximum safety.

(U) As long as personnel avoid the downwind side of the leak and are cognizant of the safety rules, the hazard risk resulting from leaks on the order of 10^{-4} scim will be low. In any event, safety clothing and breathing gear will be required if downwind work is necessary for longer than a few minutes.

5. SEAL AND SEAT DESIGN

a. Seal Analysis and Design

(1) Mode of Interface Deformation

(U) The compatibility requirement eliminates plastics and elastomers from consideration as the primary seal material. Since metals must be used, the two surfaces to be forced together to form a seal may be either of the following:

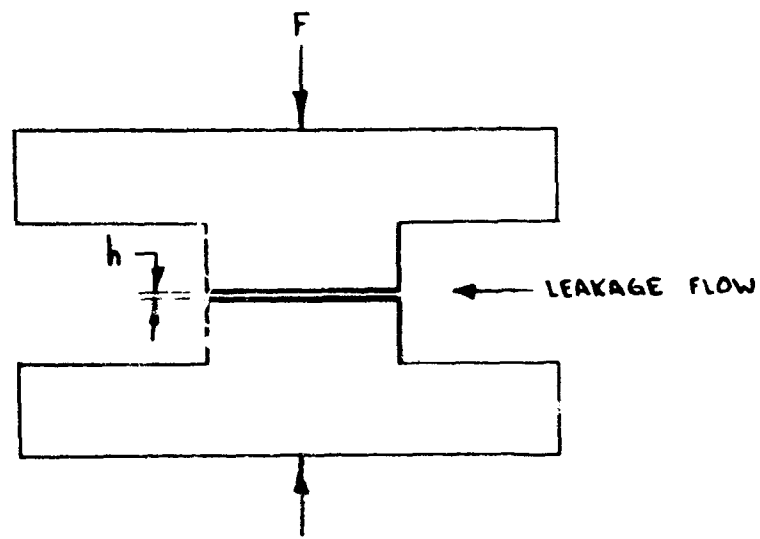
- (a) Superfinished to produce an elastically deformed sealing interface.
- (b) Machine finished to a good surface and plastically deformed to produce a sealing interface.

(U) The configuration for Case 1 is one of two flat mating surfaces as shown in Figure 16a. When pressed together, sufficient force must be applied to elastically deform the peaks of roughness and waviness asperities so that leakage through any gaps and voids which remain is reduced to an allowable value. The low viscosity of the oxidizer vapor and helium leak-test medium to be used in the quick disconnect requires that remaining voids and gaps be extremely small, but, due to the nonhomogeneous nature of metals at the microscopic level, such gaps cannot be completely eliminated.

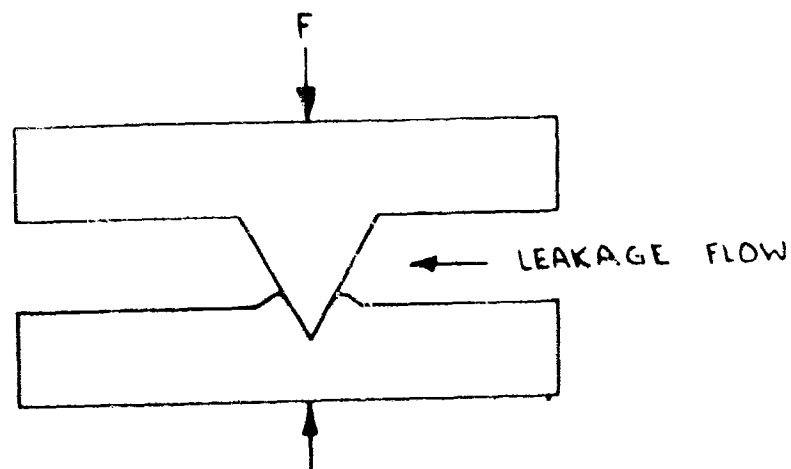
(U) The configuration for Case 2 is a hard, wedge-shaped surface biting into a soft gasket as shown in Figure 16b. When first contact is made, a high stress concentration develops at the contact points, and plastic deformation results. An increase in load causes plastic shear flow within the soft gasket, and the surface contact area around the circumference of the seal increases directly as the applied load. This bulk shear flow of the softer material results in close conformation of the mating surfaces whenever contact has been made. As in Case 1, however, even the softer metals are not homogeneous, and leakage still results after the apparent contact area forms a complete line of contact at the sealing interface. Further application of load increases the width of contact, decreasing the probability of interconnecting voids and thus decreasing leakage.

(U) An analytic comparison of these two approaches shows leakage characteristics for various applied loads. The model for this analysis is shown in Figure 17, and the leakage equation is the same for both cases:

$$\text{Leakage} = Q = \frac{\pi D(P_i^2 - P_o^2)}{12\mu P_o L} h^3 + \frac{1.06\epsilon\lambda_o(P_i + P_o)}{\mu L} D h^2 \quad (3)$$

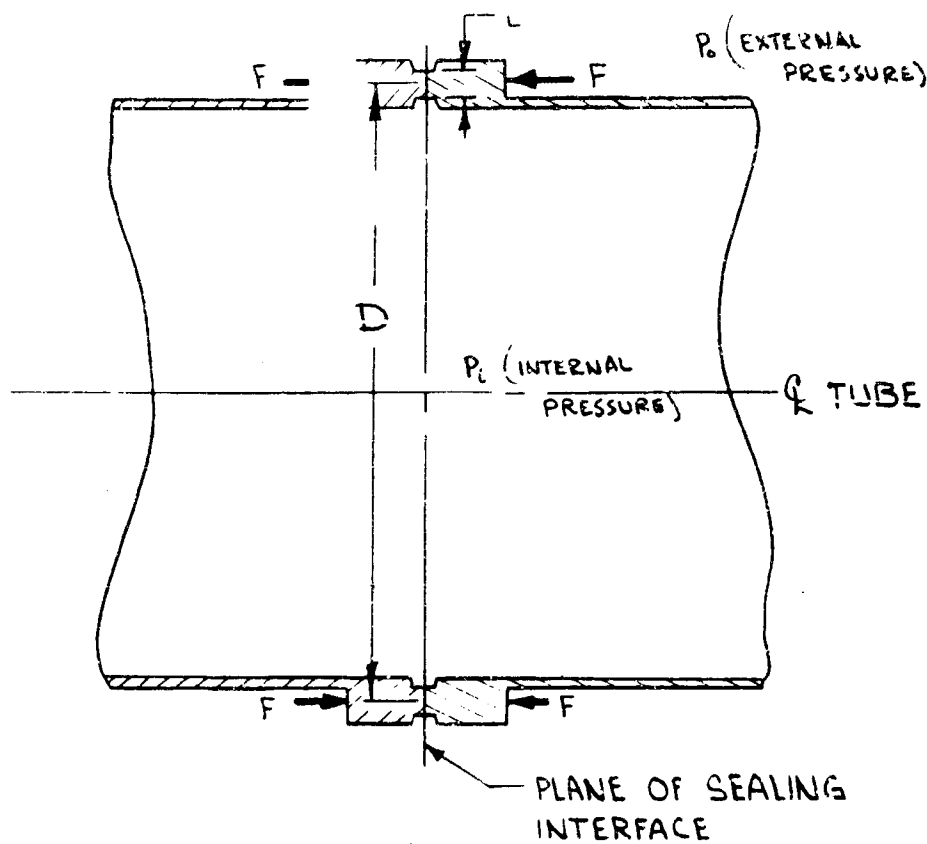


(a) FLAT MATING SURFACES



(b) WEDGE MATING WITH A
FLAT SURFACE

Figure 16. Seal Interface Configurations Model for Primary Seal Leakage Analysis



F = The applied sealing load in pounds per inch of circumference

D = Mean diameter of the seal in inches

L = Width of seal in inches

P_i = 53.7 psia

P_o = 14.7 psia

Figure 17. Model for Primary Seal Leakage Analysis

where:

Q = leakage rate (in.³/sec at STP)

D = mean diameter of seal; L = width of seal
su

P_i = internal pressure psia = 53.7

P_o = external pressure psia = 14.7

μ = viscosity of leaking medium

$$\eta = 2.8 \times 10^{-9} \frac{\text{lb-sec}}{\text{in.}^2}$$

ϵ = correction factor, 0.9 for a single gas

λ = molecular mean free path, 13×10^{-6} in.

h^3 = conductance parameter, in.³.

(U) The conductance parameter is a measure of the effective separation distance of two sealing surfaces.

(a) Determination of Leakage for Case 1

(U) Material properties for the two surfaces are assumed to be similar to 17-4 PH steel, and seal width (L) is assumed to be 0.006 in.

(U) The empirical data developed in Reference 5 are used to determine the conductance parameter. A modified stress ratio is defined as:

$$\text{Stress Ratio} = \frac{\frac{F^{2/n'}}{A_A}}{\sigma_m} \quad (4)$$

where:

F = applied load lb/in.

A_A = apparent contact area = 0.006 in.²/in.

σ_m = Meyer stress for the material = 238,000 psi

n' = Meyer index for the material = 2.2

then: Stress Ratio = $0.0007 (F)^{0.91}$

(U) Using this definition of modified stress ratio, Figure 2-47 of Reference 5 "Design Criteria for Lapped and Polished Surfaces," is replotted for the range of loads of interest in the QD coupling primary seal and appears as Figure 18 of this report.

(U) The leakage Equation 1 is used, together with the conductance parameter from Figure 18, to express leakage versus applied sealing load; results are plotted in Figure 19.

(b) Determination of Leakage for Case 2

(U) Material for the soft (gasket) surface is assumed to be similar to 1100-0 aluminum. Material for the wedge is assumed to be as hard or harder than 1141 steel.

(U) As sealing load is applied, plastic deformation of the gasket causes the apparent contact area to increase. If the surfaces were perfect, leakage would approach zero at low sealing load. In the actual case, however, the load must be increased until the wedge cuts deep enough into the soft metal to close all gaps caused by waviness and roughness asperities.

(U) Tests have been conducted at Illinois Institute of Technology (Reference 5) which indicate load versus contact area relationships. The load versus leakage tests conducted and reported by the same source are so similar to the Case 2 model that the results can be used directly and are repeated here as Figure 20. Conditions for the load-leakage test were:

$$P_i - P_o = 39 \text{ PSI}$$

Test medium = helium

Surface finish -- both surfaces were
lathe turned with 0.003-in. spiral ridge

$$\sigma_m \text{ -- Meyer Stress, wedge} = 308,000 \text{ psi}$$

$$\sigma_m \text{ -- Meyer Stress, gasket} = 49,000 \text{ psi}$$

Comparison of Figures 19 and 20 shows that with an applied load of 200 lb/in., the desired leakage rate of less than 10^{-4} SCIM can be obtained by plastic deformation of the interface (Case 2) or by elastic deformation with a surface finish equal to or better than 1 μ in. (Case 1).

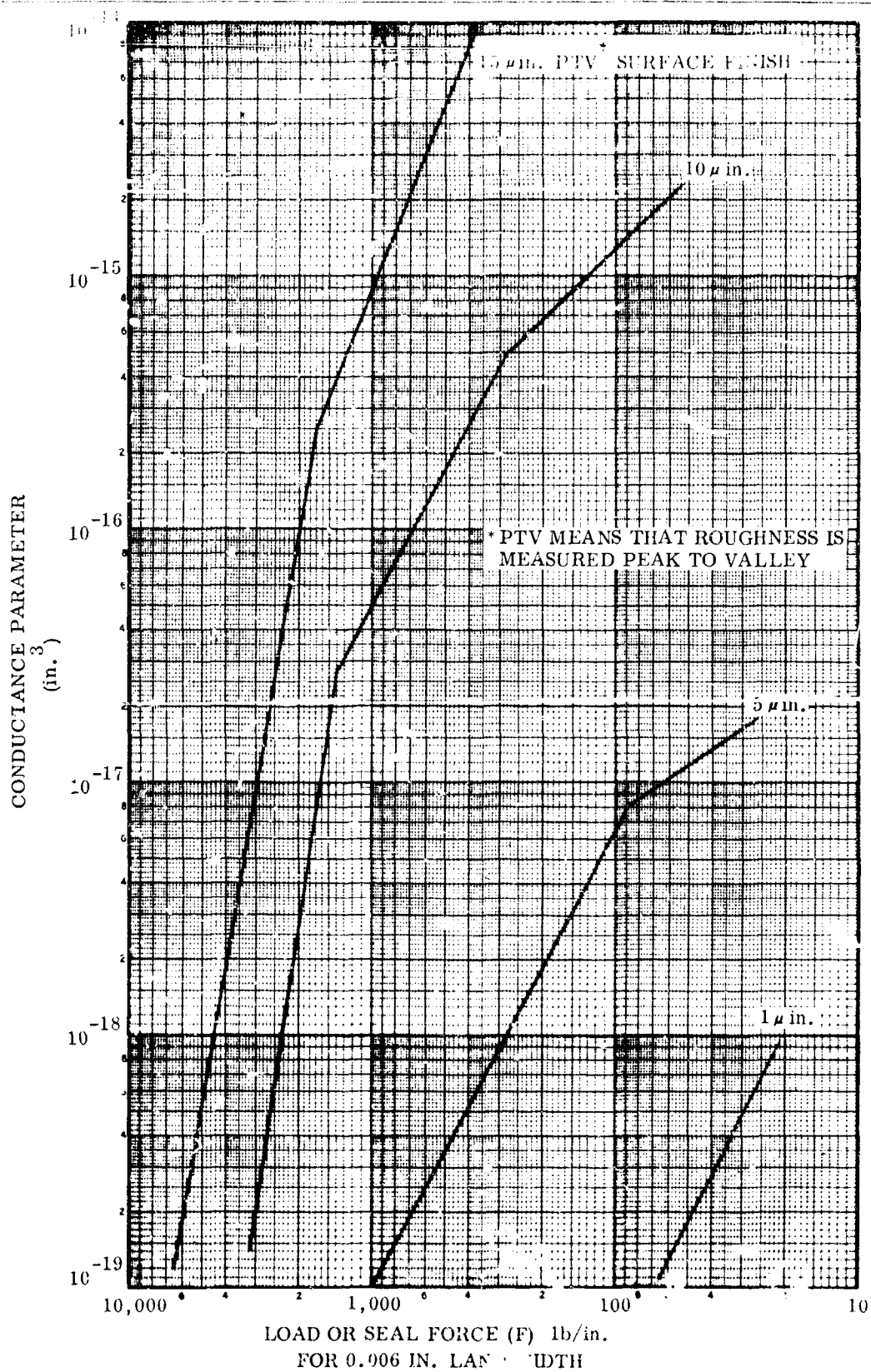


Figure 13 Conductive Versus Load for Case 1

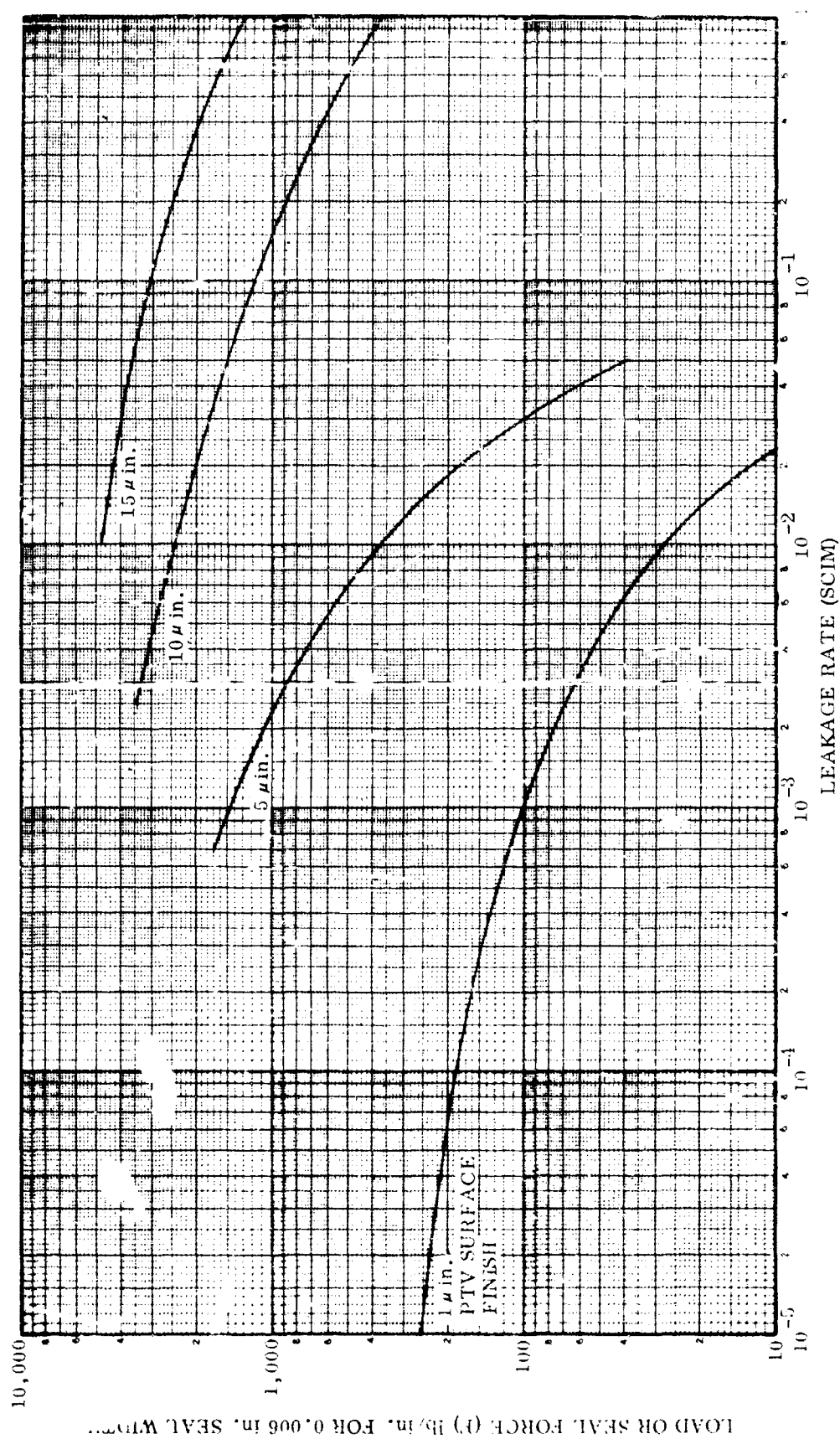


Figure 19. Estimated Helium Leakage for Case 1

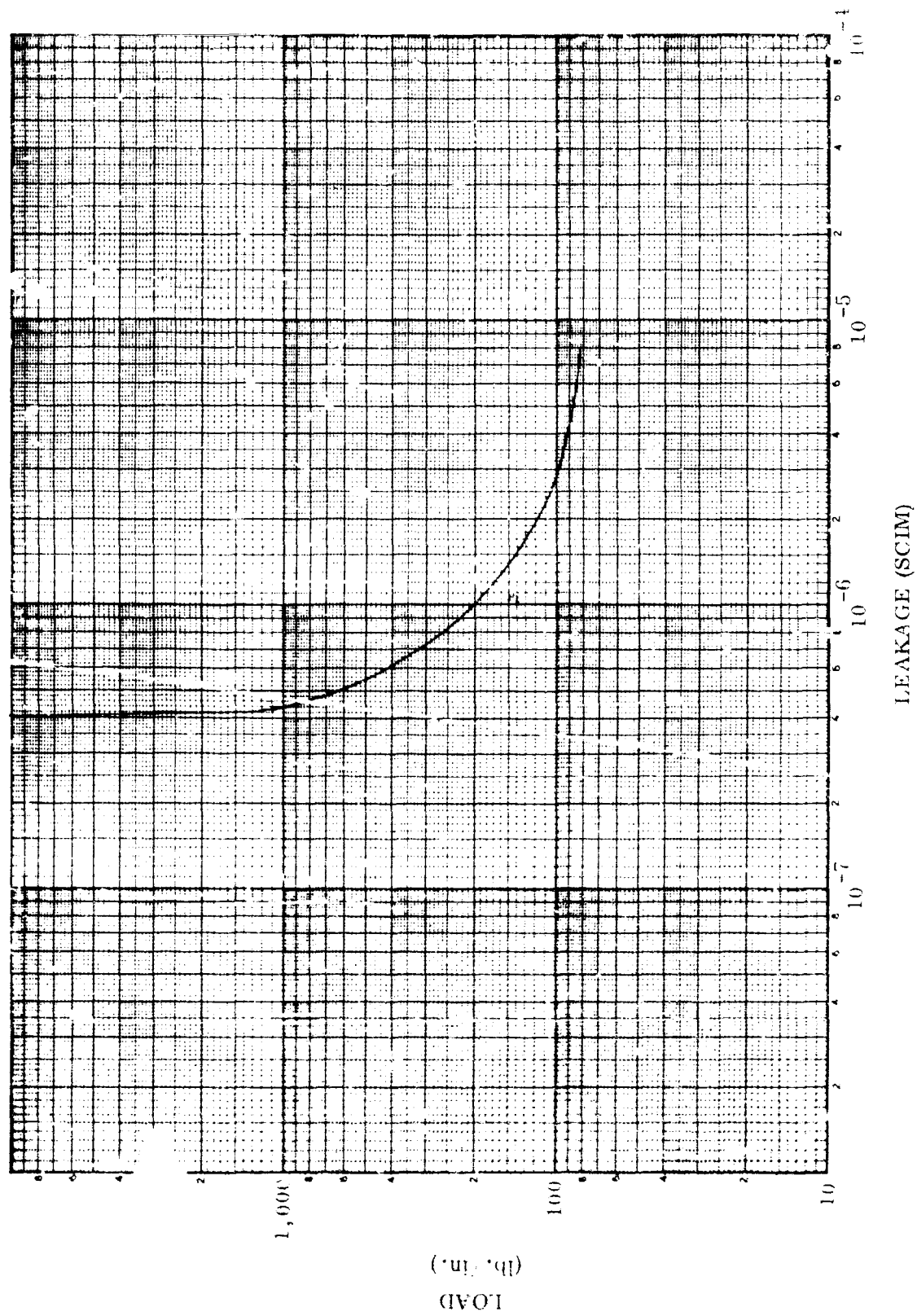


Figure 20. Estimated Helium Leakage for Case 2

(U) The superfinished surfaces of Case 1 have several disadvantages. They are difficult and expensive to fabricate, vulnerable to handling damage during transport and assembly, and susceptible to surface damage by corrosion. Further, the macroscopic geometry of the sealing surfaces must be held to extremely close tolerances, flatness, parallelism, etc., and warpage of sealing surfaces must be prevented.

(U) Correspondingly, the wedge shaped surfaces of Case 2 are not as easily damaged as the superfinished surfaces since the actual sealing surface is generated by shear flow of the softer material during coupling.

(2) Interface Geometry

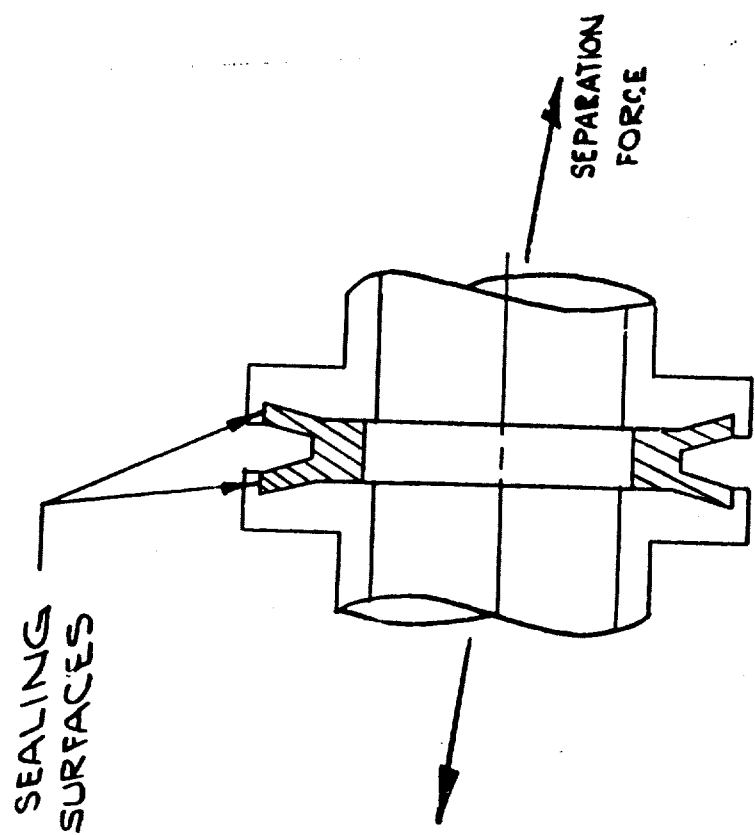
(U) The two basic choices available are flat mating surfaces and wedge-shaped surface mating against a flat surface. These are both axial seals as shown in Figure 21. A third choice, the radial sealing method used in the AF-RPL "bobbin seal" would be simple and effective, but may jeopardize a clean separation. Plastic strain of the seal material at the radial interface (Figure 21) does not relax when axial loads are removed and a separation force must be applied to shear the deformed seal material.

(U) Flat surfaces (Figure 16a) require that a superfinish be manufactured on both halves of the seal and that these surfaces be maintained through assembly, handling, and installation of the connector.

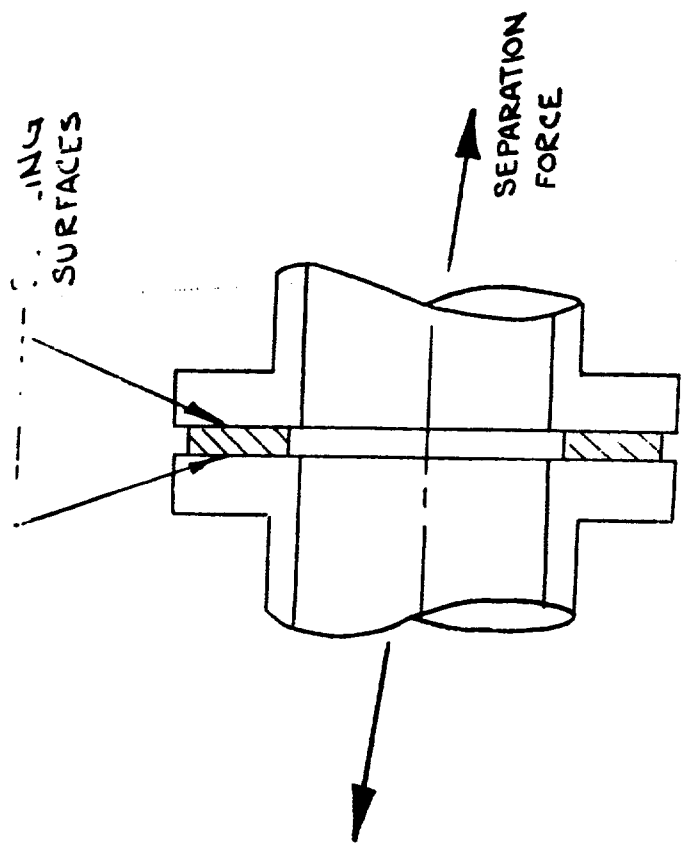
(U) The wedge mating against a flat surface (Figure 16b) provides the required concentration of stress to deform the roughness of a standard machined surface with minimum loads. It is essential however, that the soft surface be protected from handling damage until ready for use. It may have to be replaced before each recoupling. When these conditions are met, the wedge and deformable surface concept appears to meet all the requirements for quick-disconnect coupling seal, although other concepts may be equally appropriate.

(3) Structural Configuration

(U) If the required sealing interface loads can be applied and maintained as the loading and temperature environments vary, the structural configuration for this application is relatively unimportant. Oxidizer transfer will be at low pressures, which eliminates any advantage of pressure activated seals whose structural shape is most significant at pressure in excess of 2,000 psi.



RADIAL "BOBBIN" SEAL



AXIAL SEAL

Figure 21. Axial and Radial Sealing Methods

(U) During final design of the coupling seal, analysis of seal shape will be required to relate the deformation of the seal structure to the load acting at the interface. For the case of a plastically deformed gasket, this includes determination of load versus deflection and optimum gasket thickness.

(U) Several commercially available seals which provide the type of deformation necessary for sealing flat mating surfaces (Case 1) are shown in Figure 22. The surface finish on these commercial seals is approximately 5μ in., and further surface finishing of the seals, if practical, would be necessary to provide the desired load-leakage characteristics. Figure 22d shows a soft metal gasket plastically deformed between hard metal wedges for sealing (Case 2).

(4) Material Properties

(U) All components of the primary seal must be compatible with dynamic liquid oxidizer. To ensure the desired plastic flow characteristics, it may be necessary to select a material such as aluminum for the soft gasket. Aluminum, however, has a moderate corrosion rate with hydrogen fluoride. Thus, extreme caution must be taken to preclude the induction of moisture through the sealing interface during periods subsequent to passivation or oxidizer flow.

(5) Hysteresis

(U) When two surfaces are pressed together under load as a seal, a given leakage may result. When the load is released, little change in leakage rate is noticed until the load is sufficiently relaxed, whereupon a significant increase in leakage occurs. This lag in leakage increase as load decreases (hysteresis) is necessary to assure good seal performance when thermal gradients or dynamic structural stresses cause seal load relaxation. The maximum allowable load relaxation (without significant increase in leakage) depends on the maximum applied stress. Figure 23, taken from Reference 5, shows that a 50 percent reduction in the loads for the previously examined Case 2 can be effected with less than 50 percent increase in leakage. It is also noted from the same reference that if the surfaces are disjoined and rejoined in exactly the same position, full plasticity and, thus, good sealing will result if the sealing load is increased five percent over the original load.

(6) Handling Damage

(U) A seal design may be clearly optimum for the ideal conditions present in a laboratory. A good seal design, however, must perform satisfactorily after being handled and assembled by experienced technicians.

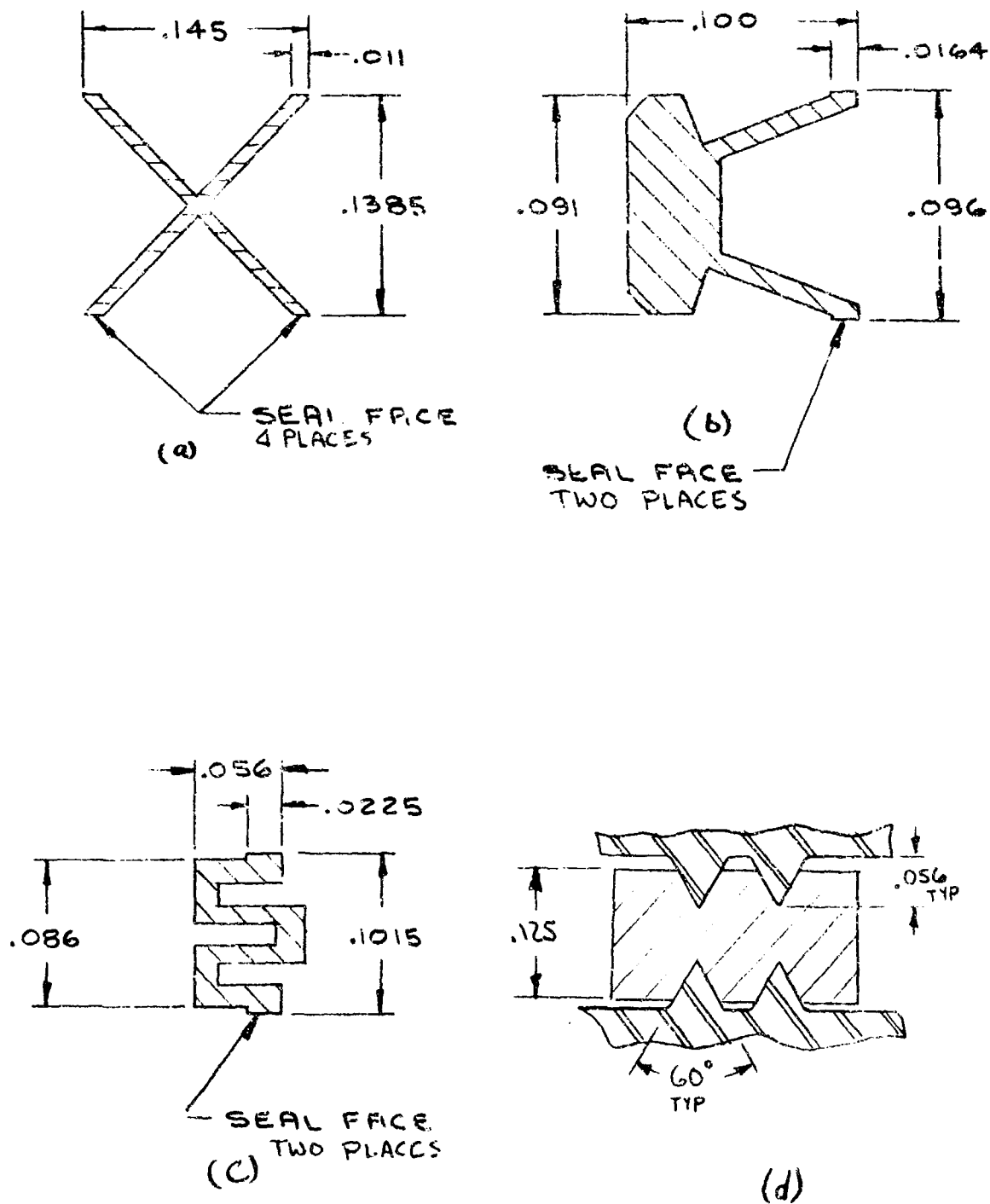


Figure 22. Commercially Available Seal Shapes

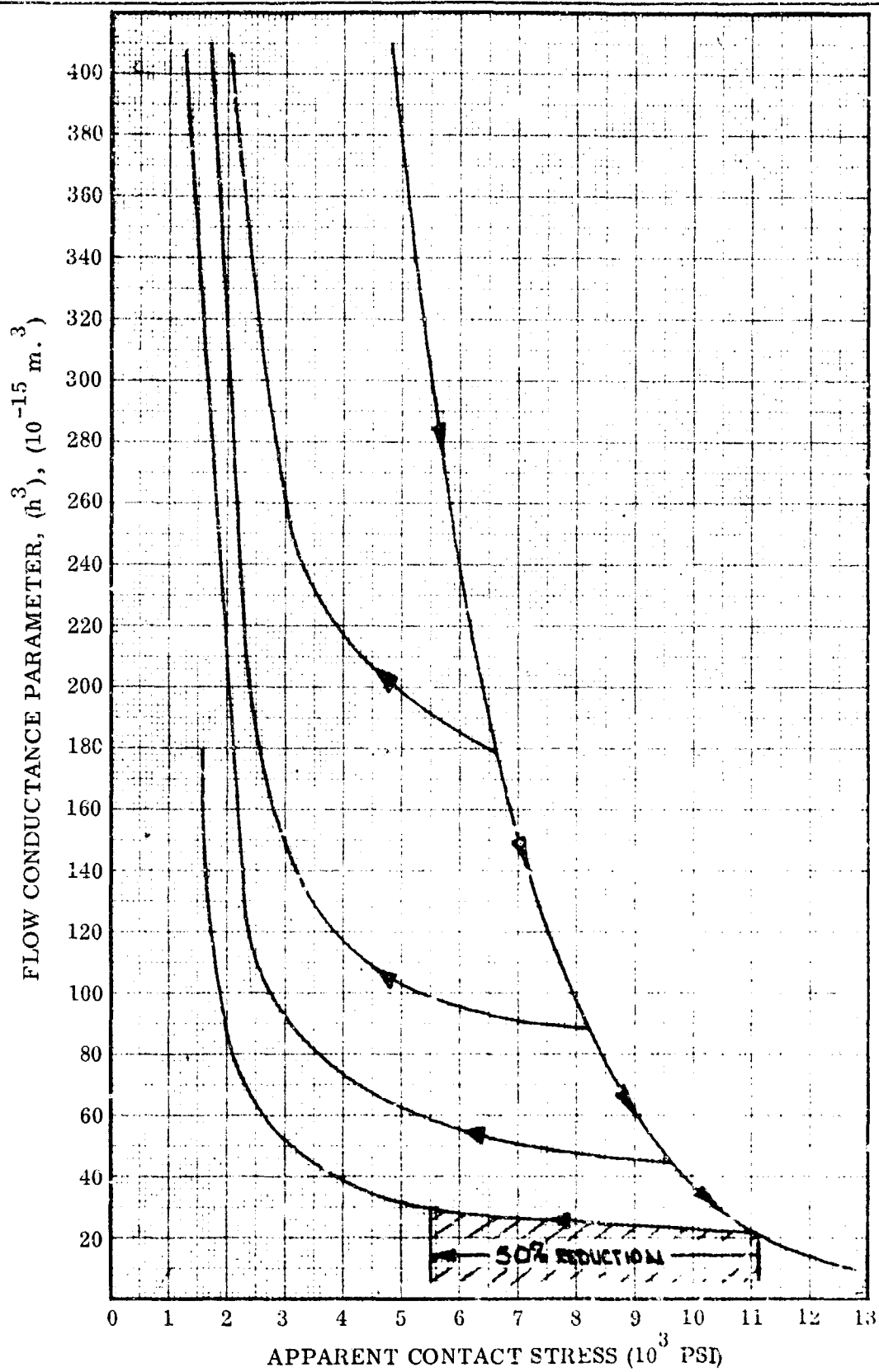


Figure 23. Hysteresis Effects for Aluminum-Steel Interface

(U) If the surface hardness is at least DPH = 400 (diamond point hardness), a 220-lb load on the Vickers diamond will cause an impression approximately 0.004-in. deep and 0.020-in. wide. It is unlikely that a greater load would be applied or that a harder, sharper instrument would be used in damaging the surface. Damage resulting from careless handling procedures may take the form of a notch in the wedge of shape similar to that of the Vickers diamond impression. By using this as the model for leakage of a damaged surface, the hydraulic radius (ratio of flow area to the wetted perimeter) resulting from a notch 0.020-in. wide and 0.004-in. deep is 0.001 in.

(U) For the case of a liquid fluorine seal, the damaged area may allow gaseous leakage if sufficient heat is available to vaporize the liquid as it flows through the leak path or, if the coupling body is prechilled, liquid leakage may result. In either case, gas or liquid temperature is assumed to be 150°R, and internal pressure 100 psia.

(U) Gaseous fluorine flow through an orifice, assuming choked flow, is:

$$\dot{W}_g = CAP_i \sqrt{\frac{g\gamma}{RT_i} \left(\frac{2}{\gamma+1} \right)^{\frac{\gamma+1}{\gamma-1}}} \quad (5)$$

where:

\dot{W}_g = gaseous flow rate lb/min.

$A = (0.001)^2 \pi \text{ in.}^2$

P_i = internal pressure = 100 psia

$R = \pi 90 \text{ in. / } ^\circ\text{R (for fluorine)}$

$T_i = 150^\circ\text{R}$

g = gravitation conversion factor
 $= 1.39 \times 10^6 \text{ in./min.}^2$

γ = ratio of specific heats for fluorine = 1.4

C = nozzle discharge coefficient = 0.95

then:

$\dot{W}_g = 0.0087 \text{ lb/min.}$

(U) Liquid flow through the same orifice, assuming turbulent flow conditions, is:

$$\dot{W}_L = WCA \sqrt{2g \frac{\Delta P}{w}} \quad (6)$$

where:

$$w = LF_2 \text{ density} = 0.054 \text{ lb/in.}^3$$

$$\Delta P = 85.3 \text{ psi}$$

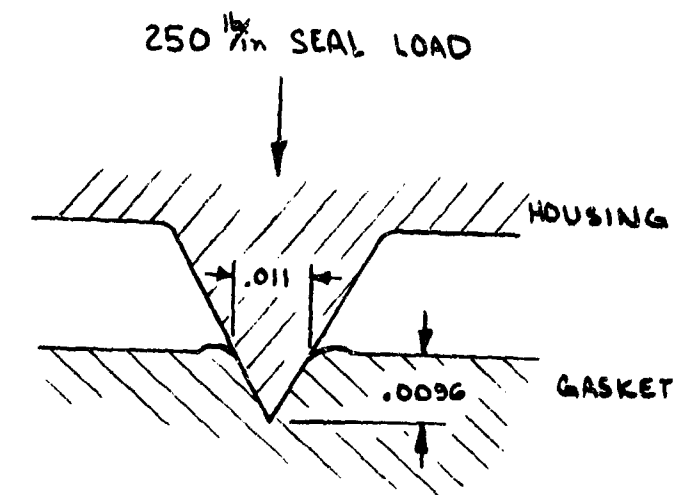
then:

$$\dot{W}_L = 0.00285 \text{ lb/min.}$$

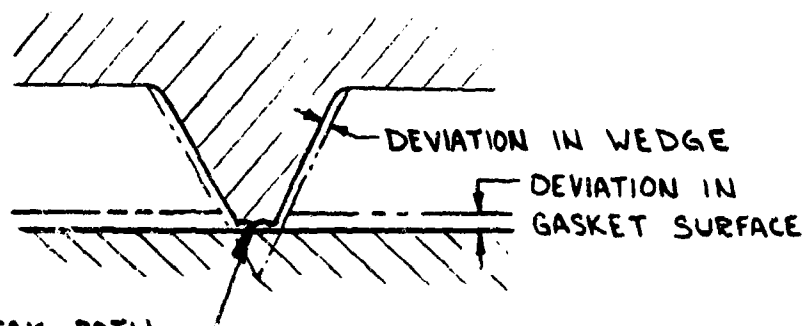
(U) The density of fluorine gas at ambient conditions is $5.62 \times 10^{-5} \text{ lb/in.}^3$ so that the liquid leak corresponds to 50.8 standard cubic inches per minute (SCIM) and the gas leak corresponds to 15.7 SCIM. The plastic deformation of the soft metal gasket by a 60° wedge as shown in Figure 24 will tend to reduce the size of leak path caused by handling damage so these leakage estimates are somewhat larger than would be experienced in the real case.

(U) It should be mentioned at this point that fabrication of a circular wedge surface with the apex in a flat plane is difficult. Test surfaces of Reference 5 typically varied 0.002 in. from the flat plane. Closer control of manufacturing methods will result in a probable tolerance of $\pm 250 \mu\text{in.}$ from a flat plane. The variation of the wedge surface, local roughness, variation of thickness of the deformable gasket, and handling damage to either surface can reduce the penetration of the wedge and result in excessive leakage. For this reason, it is recommended that a redundant wedge be used to eliminate the possibility of leakage caused by the noted local surface imperfections.

(U) Special handling is required for soft metal gaskets. Because clean replacement parts must be available, only a small additional cost will be incurred to provide an individual rigid container for each gasket. If the container remains unopened until just before installation, a clean, undamaged gasket will be assured.



(a) NORMAL WEDGE PENETRATION
FOR 250 $\frac{1}{16}$ SEALING LOAD



POSSIBLE LEAK PATH
IF WEDGE IS DAMAGED

(b) ADVERSE TOLERANCE BUILDUP AND
A SEVERELY DAMAGED WEDGE

Figure 24. Plastic Deformation of a Soft Metal Gasket

(7) Seal Housing Deformation

(U) In addition to the structural forces applied by the missile and AGE to the connector, the variable thermal environment must be considered. The major effects of the thermal environment on the connector are as follows:

- (a) Reduction in contact stress at the seal as a result of creep or relaxation of the attachment mechanism.
- (b) Differential thermal expansion resulting from differences in material of the seal and housing assembly and from temperature gradients during initiation of flow of cryogenic fluids.

(U) In general, the connector mechanism must be designed to provide the required sealing loads to the seal interface at all times. The mechanical and thermal effects on the final housing design should be evaluated to assure that thermal deformations acting in conjunction with mechanically induced loads will not cause relaxation of the sealing loads beyond the 50 percent allowed by hysteresis of the seal material.

(8) Primary Seal Failure

(U) After the coupling is connected and sealing load has been applied to the seal interface, a leak check must be accomplished. Helium or nitrogen can be introduced to the volume between the vehicle and AGE shutoff valves and system pressure applied. The coupling will probably not be separated again until propellants have been loaded and the vehicle is committed to lift off the launch support structure. If leakage should develop during the interim, the possible causes are as follows:

- (a) Relaxation of the sealing load.
- (b) Corrosion of the interface.
- (c) Relative motion between the sealing surfaces.
- (d) Structural failure of the seal material.

(9) Structural Failure

(U) Structural failure of the seal material is not probable if an adequate quality control procedure has been followed in selection of the material and during the manufacture of the coupling.

(10) Relative Motion

(U) If the two opposing surfaces which sandwich the gasket are allowed to move laterally with respect to each other, the

possibility of breaking the seal exists. Either the gasket material will deform or sliding will occur at the interface. If sliding occurs, the microscopic contact is lost, probably opening a leak path. Transverse structural loads should not be carried by the seal if the coupling is properly designed, but differential thermal expansion resulting from dissimilar materials, gasket shape, or severe temperature gradients could cause relative motion. When the thermal gradient disappears, the seal may return to a different condition of stress equilibrium, with loads sufficiently reduced to cause increased leakage.

(U) Dynamic relaxation of stress, caused by superposition of an alternating stress on a highly stressed region, could be significant if vibration and/or shock conditions are encountered during propellant transfer and valve shutoff. Leakage then results, as previously discussed, when sealing loads are reduced and surface contact is lost.

(U) Because of the unknowns involved in the dynamic and thermal stress relaxation cases, it is recommended that a closed cavity external to the primary coupling seal be designed into the coupling from which a continuous or at least periodic sample can be taken to verify that there is no seal leakage before and during propellant transfer. This cavity can be continuously evacuated or purged and the vent gas disposed of safely in the event of leakage. If the vent gas is monitored, any fluorine leakage which might develop can be detected in time to avert a failure. A design concept is shown in Figure 24.

(11) Summary

- (a) The primary seal must be made of metal because it is exposed to liquid propellant flow.
- (b) The primary seal must be designed for a leak rate less than 10^{-4} cc/sec when tested with ambient gaseous helium at the expected system pressure. This value satisfies the "no-leak" specification of the Preliminary MANSAT Oxidizer QD Requirements.
- (c) Study of surface finish and load required to form a seal indicates that plastic deformation of one surface is the best method of sealing for this application.
- (d) For the 100 to 500 lb per in. range of loads available for this application, a hard wedge acting on a soft aluminum surface provides an excellent seal.
- (e) The circular wedges must be made of the hardest compatible material available. Hard materials will minimize mechanical handling damage.

- (f) The possibility of liquid leakage will be reduced by the use of a redundant wedge.

b. Seat Analysis and Design

(U) During the initial QD evaluation study, design and analysis were carried out to determine the capabilities of a poppet check valve, designed as an integral part of the QD vehicle half. Several concepts for the valve design were evolved and evaluated. One concept utilized spherical poppets and seats for the valve and another utilized spherical poppets and seats for the valve and another utilized flat poppets and seating surfaces. Conical mating surfaces for the valve were considered, but the conical concept was eliminated early because it appeared to be more vulnerable to the buildup of adverse tolerances during fabrication than either the flat or spherical configuration. Figure 25 is a concept of a 2-in. quick disconnect showing probable arrangement and size of poppet and seat.

(1) Leakage Analysis

(U) The following paragraphs discuss a preliminary analysis to estimate the leakage of a flat seat made from Inconel X with yield strength (y) in the range of 95,000 to 110,000 psi. In this analysis, surfaces of the poppet and seat are considered to be flat within 6μ in. and smooth within 1μ in. peak to valley (PTV). Waviness is considered to be surface variations with a wave length greater than 0.035 in. Also, the poppet must be angularly unrestricted so the surfaces will be parallel when closing. Case 2 is a baseline with the above restrictions; Case 1 and Case 3 will show the effect of changing surface finish variables.

(U) The Reference 6 method will be used to predict leakage which requires use of the surface finish variable θ , a measure of the average asperity slope as well as h , the effective asperity height. Because slope is difficult to measure directly, this may be represented by:

$$\theta = \frac{2h}{\text{peak-to-peak wave length}} \quad (7)$$

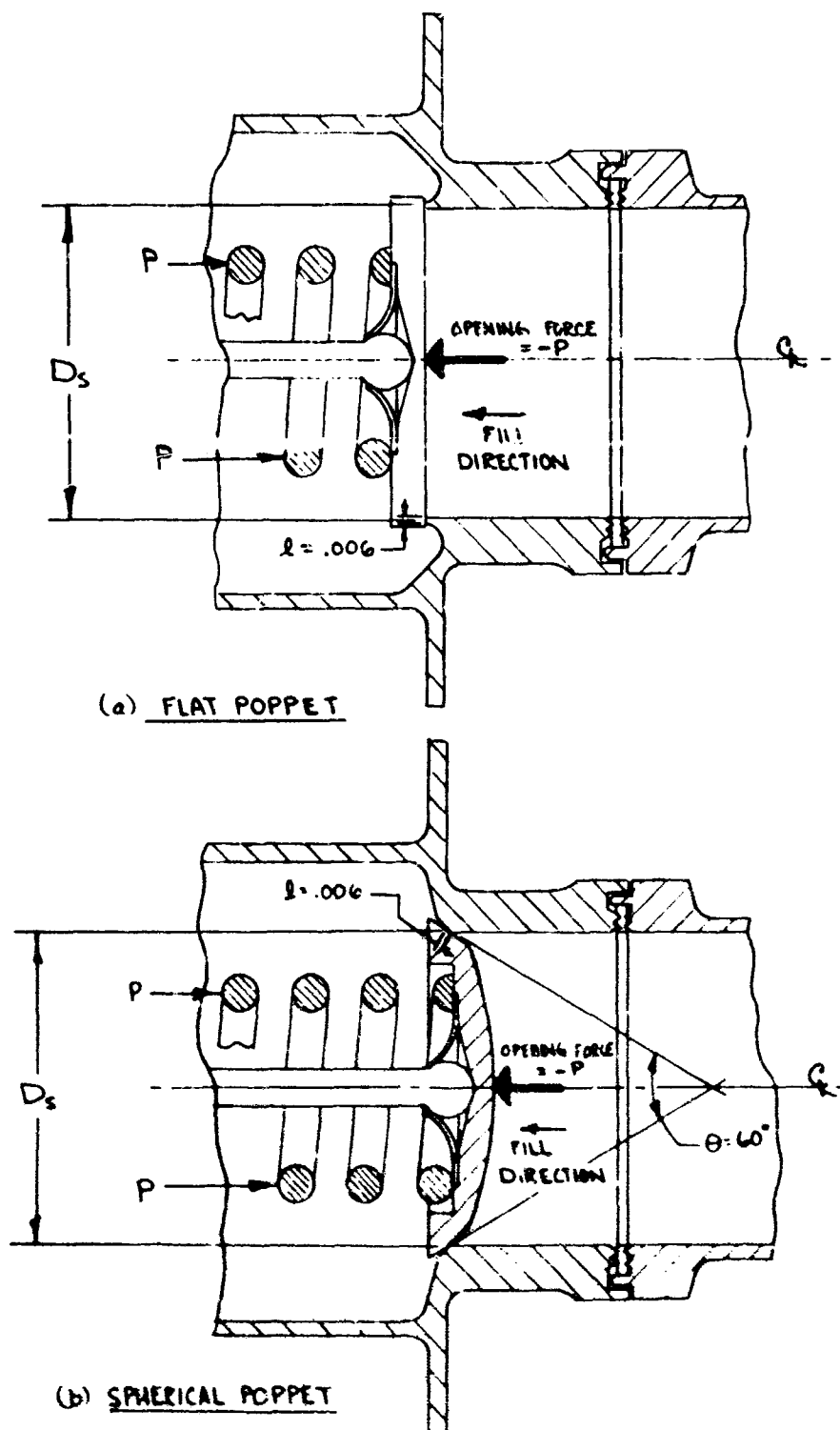


Figure 25. Propellant Check Valve Concepts

$$Q = \frac{4.71 D_B (P_1^2 - P_2^2)}{\mu l T} \left\{ 1.36 \left[h - \frac{3}{4} \left(\frac{36 S^2 h^3}{\phi^2} \right)^{1/3} \right] \right\}^3 + \frac{1.42 \times 10^5 D_B}{l} (P_1 - P_2) \sqrt{\frac{R}{T}} \left\{ 1.22 \left[h - \frac{3}{4} \left(\frac{36 S^2 h^3}{2} \right)^{1/3} \right] \right\}^2 \quad (8)$$

D_s = seat mean diameter = 2.25 in.
 P_1 = internal pressure = 64.7 psia (max.)
 P_2 = external pressure = 14.7 psia (max.)
 l = seat land width = 0.006 in.
 μ = fluid viscosity = $4.72 \times 10^{-11} \frac{\text{lb-min.}}{\text{in.}^2}$
 (for helium)
 R = gas constant = $4632 \frac{\text{lb.min.}}{\text{lb}^\circ\text{R}}$
 (for helium)
 T = absolute temperature = 540°R
 α = elastic constant = $2(1 - \nu^2)/E = 5.87 \times 10^{-8}$
 $\text{in.}^2/\text{lb}$ (for Monel)
 S = apparent contact stress = $\frac{P}{\pi D_s l}$
 h = PTV roughness of one surface
 h' = PTV waviness of one surface
 ϕ = average asperity slope (roughness)

ϕ' = average asperity slope (waviness)

l = width of seal contact

(U) Because minimum weight and complexity for the QD vehicle half is a design goal, the spring force required for sealing must be kept to a minimum. For the 2-in. coupling of Figure 25, the range of available load from a simple spring is from 100 to 600 lb, resulting in an apparent sealing stress of 2,500 to 14,000 psi. Listed in Table III and plotted in Figure 26 are the calculated values for helium leakage versus apparent contact stress for the following surface conditions:

Case 1 -- Roughness $h = 1\mu$ in AA = 3μ in PTV

$$= 1/4^\circ = 1/229 \text{ radians}$$

Waviness $h' = 2-1/2\mu$ in PTV

$$\phi' = 1.43 \times 10^{-4} \text{ radians}$$

Case 2 -- Roughness $h = 1/3\mu$ in AA = 1μ in PTV

$$\phi = 1/4^\circ = 1/229 \text{ radians}$$

Waviness $h' = 12\mu$ in PTV

$$\phi' = 6.86 \times 10^{-4} \text{ radians}$$

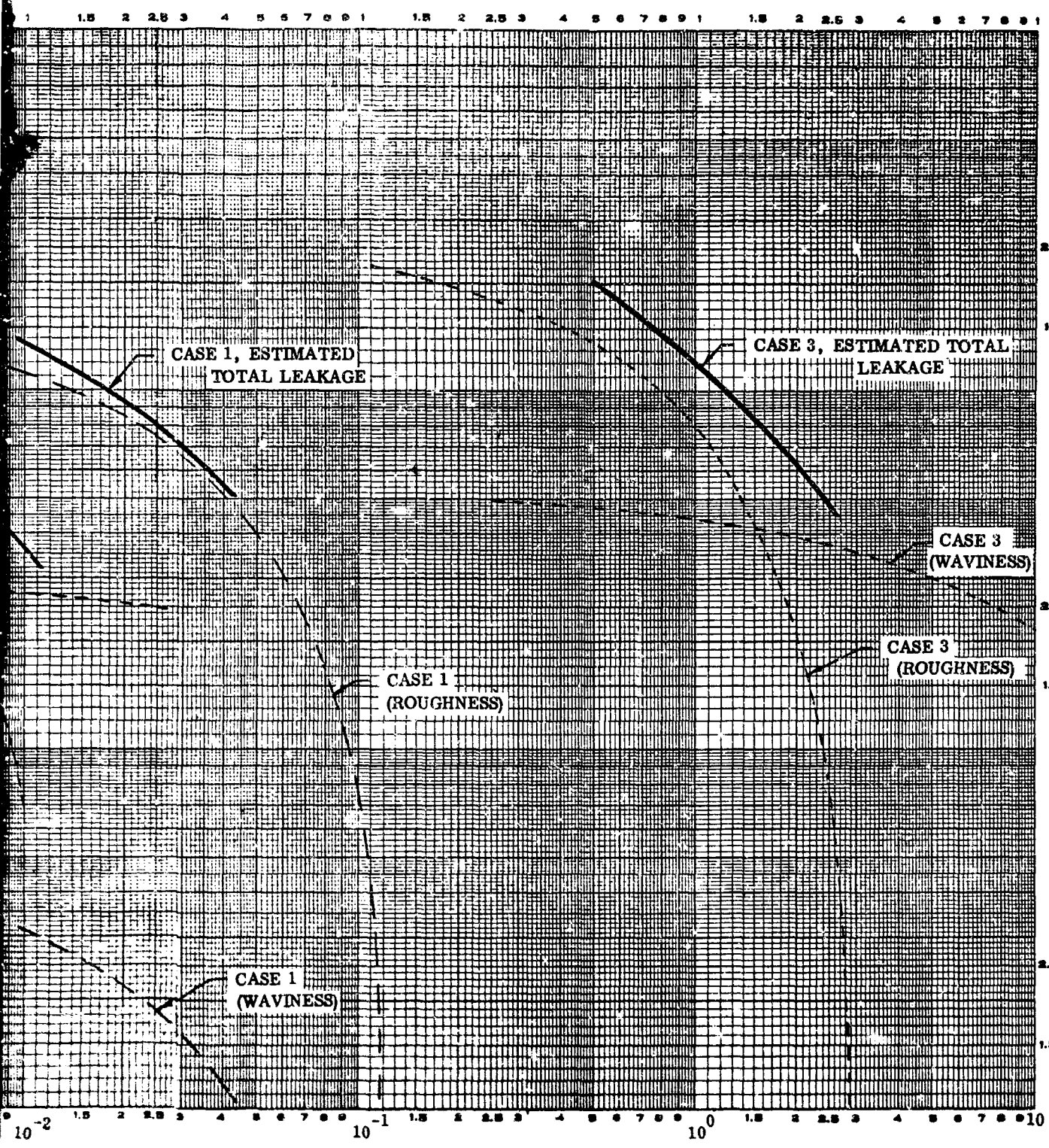
Case 3 -- Roughness $h = 4\mu$ in AA = 12μ in PTV

$$= 1/2^\circ = 1/115 \text{ radians}$$

Waviness $h' = 24\mu$ in PTV

$$\phi' = 13.72 \times 10^{-4} \text{ radians}$$

(U) The analysis indicates that a well designed check valve with a good poppet and seat surface finish, represented by Case 3, will result in leakage of approximately 1 SCIM. The best known (Reference 6) surface finish, represented by Case 2, will result in a leakage rate of 10^{-2} to 10^{-3} SCIM. From the standpoint of controlling the excessive loss of liquid propellants, any of the cases investigated would be more than adequate. Should this type of valve be required to seal the oxidizer from areas inhabited by personnel, the capability of a poppet check valve is questionable, and a different valving approach is presently indicated.



Helium Leakage - (SCIM)

Leakage for Various Surface Conditions

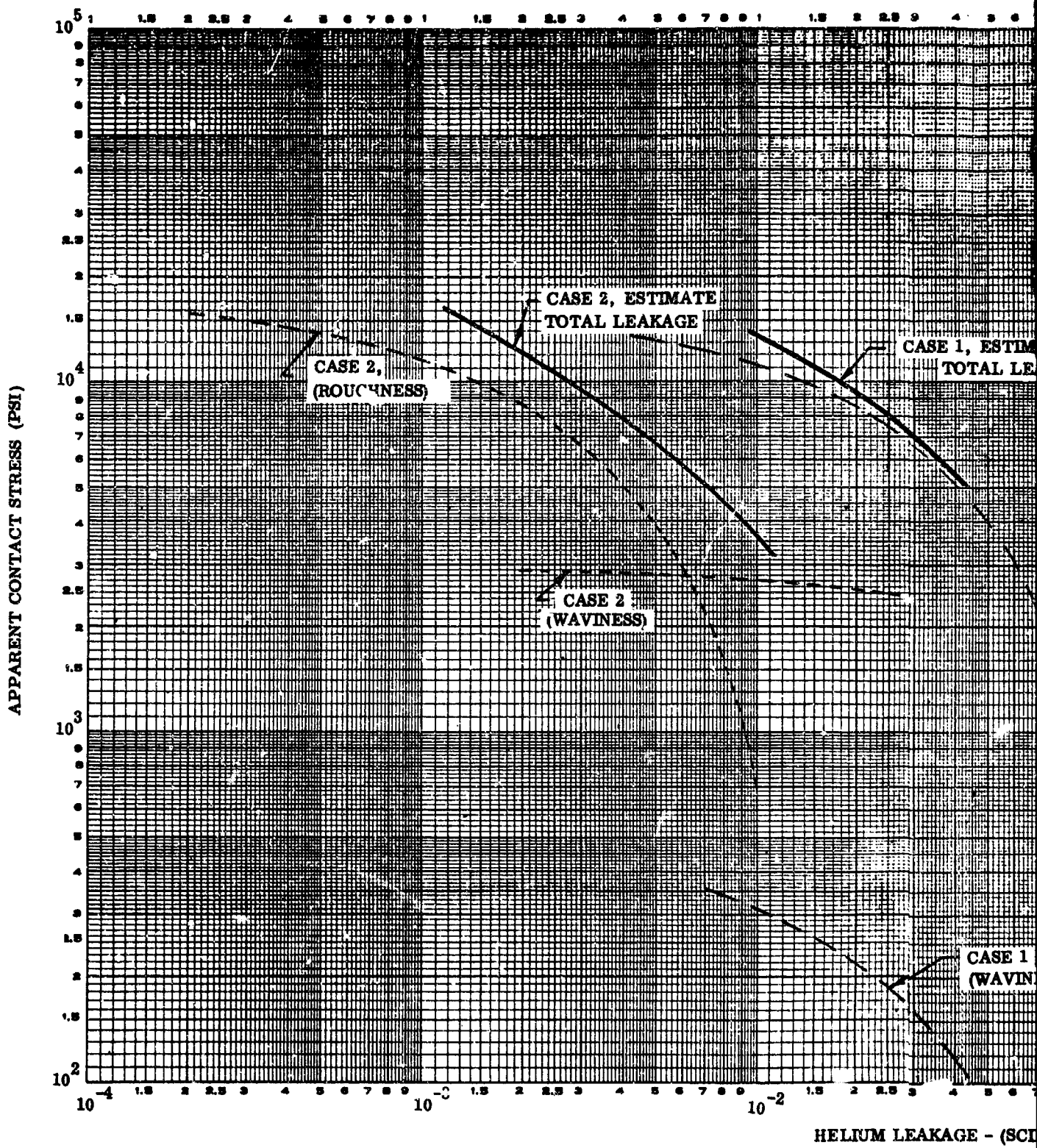


Figure 26. Stress Versus Leakage for Various Surface C

Table III

STRESS AS A FUNCTION OF LEAKAGE FOR VARIOUS SURFACE CONDITIONS

	h(μ in. PTV)	ϕ (Radians)	h' μ in. (PTV)	ϕ' (Radians)	Asperity type	S (psi)	Q (SCIM)
Case 1	3	$\frac{1}{229}$			Roughness	10,000 3,000 -0-	1.36×10^{-2} 5.88×10^{-2} 12.35×10^{-2}
Case 1			2-1/2	1.43×10^{-4}	Waviness	625 400 100	-0- 5.09×10^{-3} 49.50×10^{-3}
Case 2	1	$\frac{1}{229}$			Roughness	10,000 3,000 1,000	1.45×10^{-3} 5.92×10^{-3} 12.38×10^{-3}
Case 2			12	6.16×10^{-4}	Waviness	3,000 2,500 2,000	-0- 2.23×10^{-2} 6.53×10^{-2}
Case 3	12	$\frac{1}{115}$			Roughness	18,000 10,000 -0-	0 0.256 0.83 2.86
Case 3			24	13.72×10^{-4}	Waviness	6,000 3,000 -0-	-0- 5.12 16.32 (U)

(2) Poppet Configuration

(U) A spherical poppet acting on a narrow seat as shown in Figure 25 has several advantages over a flat seat, as follows:

- (a) The spherical poppet provides a smoother flow path for the flowing fluid.
- (b) The valve seat can be machined as an integral part of the valve housing.
- (c) The valve seat will be easier to refurbish if it should be damaged by pits or scratches.
- (d) Because of its geometry, a spherical poppet has a greater seating force than a flat poppet.

(U) The disadvantages of the spherical concept are as follows:

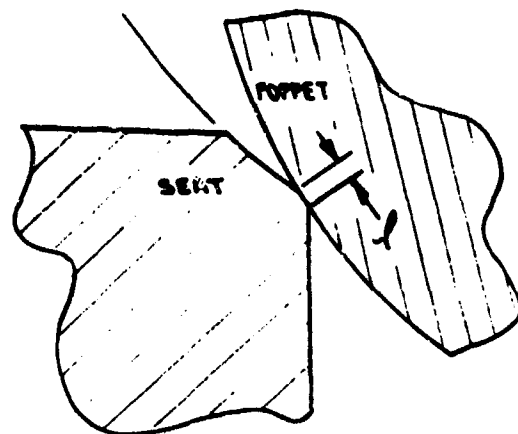
- (a) Spherical surfaces are generally more difficult and costly to produce than flat surfaces.
- (b) Measurement of roughness and waviness on a spherical surface is more difficult than on a flat surface.

(U) The spherical seat may be formed by lapping with a tool of diameter identical to the poppet or by coining with an extremely smooth hardened tool of poppet diameter.

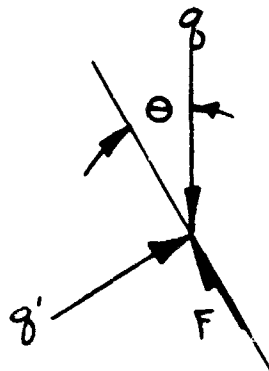
(U) Lapping surfaces to the required degree of smoothness requires special care on the part of the machinist. The tool must be constantly checked and compared against a master for wear and expansion resulting from the heat of lapping.

(U) The manufacture of precision surfaces by coining requires the fabrication of a hardened steel tool with a surface better than the desired seat surface. Experimental evidence described by Archard (Reference 7) indicates that metal surfaces pressed flat by a carefully polished hardened steel anvil assume a surface contour similar to the anvil. It was shown by electron microscopic examination that the resulting slopes of the asperities were in all cases less than $1\frac{1}{2}^\circ$ and were completely elastic in that the asperities could be pressed just flat without plastic flow. Having confidence from these results that coining will produce a satisfactory seat, the required coining load can be determined. To produce a 2.25-in. diam seat of 0.006-in. width at an included angle of 60° (assuming full plastic flow of the seat material at an apparent stress level of $\sigma_y = 300,000$ psi),

$$P = 0.006 (7.07) (300,000) \sin 30^\circ = 12,700 \text{ lb}$$



(a) SPHERICAL SEAT
ARRANGEMENT



(b) SEAT LOADING DIAGRAM

Figure 27. Spherical Seat Contact Load

(U) Contact is made at an angle to the direction of closing motion so that the interface load is increased over that of a flat poppet.

(U) The closing load q is resisted by a force q' (which is normal to the spherical surface) and a friction force, F (see Figure 27b).

$$q' = \frac{q - F \cos \theta}{\sin \theta}$$

$$F = f q' \tag{9}$$

$$q' = \frac{q}{\sin \theta + f \cos \theta}$$

If $\theta = 30^\circ$ and a coefficient of friction (f) of 0.39 is assumed, then $q' = 1.2 q$.

(U) In this case, the sealing load for a spherical poppet is 1.2 times the sealing load for a flat poppet for the same applied axial load. As noted from the above equation, the coefficient of friction between the closing surfaces influences the applied force, and, from Reference 8, the coefficient of friction has been observed to change because of the formation of a fluoride film after exposure to the oxidizer. Further information is required concerning the tenacity and toughness of the fluoride film during closing of the valve for both spherical and flat poppets.

(U) Douglas has concluded that the spherical seat design concept is best for this particular application.

6. SUMMARY OF MATERIALS COMPATIBILITY

(U) Fluorine reacts spontaneously with many materials at room temperature and, under suitable conditions, with virtually every material. No organic material is completely resistant to fluorine except carbon tetrafluoride, which is the stable end product of the reaction between fluorine and carbon. Fluorine also reacts with most inorganic materials, except the light inert gases and metal fluorides in their highest valence states. The reaction with most metals is comparatively slow at room and lower temperatures but vigorous at elevated temperatures (Reference 9). Insofar as compatibility with materials is concerned, the oxidizers may be divided into two groups, cryogenics (liquid fluorine, LF_2 , and FLOX) and storables (chlorine trifluoride, or CTF, and Compound A). In general, materials which are compatible with F_2 are compatible with FLOX, and materials which are compatible with CTF are compatible with Compound A. The metals most resistant to LF_2 are nickel, Monel, low-silicon stainless steel, aluminum, copper, and magnesium (see Appendix III for some tabulated test results). Wrought Monel is one of the most satisfactory materials for use with LF_2 . In addition to resisting LF_2 , Monel also resists attack by hydrogen fluoride (HF) which is a common contaminant in LF_2 . The corrosion resistance of metals is apparently based on the formation of fluoride films which protect the metal from further attack. However, permeability of the metal is one of the controlling factors in corrosion rate, and the fluoride coating varies with the material. The least reactive, or the most resistant metals, are the least dependent on the fluoride film.

(U) Based on information gathered and published by the Defense Metals Information Center (Reference 10) metals which should not be used in LF_2 service include low-carbon steel, cast iron, cast Monel, lead, molybdenum, tantalum, titanium, columbium, and zirconium. Almost all of the nonmetals react rapidly with LF_2 and cannot be used.

(U) Teflon is sufficiently inert for limited use. Its use must be limited to static environments, and extreme precautions must be taken to ensure that the material is free from contamination. Other nonmetallic materials such as synthetic sapphire, Norbide, and Kentanium reportedly react very slowly in F_2 and may be suitable for certain applications.

(U) The common structural metals most resistant to CTF and Compound A, roughly in the order of decreasing resistance, are nickel, Hastelloy C, Monel, copper, Inconel, aluminum, 300 series stainless steels, low-carbon steel, and magnesium. Teflon and Kel-F can be used under limited and controlled service conditions. They should never be used in service with these oxidizers when subjected to heat, shock, or flow conditions.

(U) Metals known to be unsuitable for use with CTF and Compound A include titanium, columbium, molybdenum, uranium, zirconium,

lithium, vanadium, niobium, tungsten, mercury, boron, silicon, arsenic, and antimony. Almost all of the organic compounds react with these oxidizers. Carbon and graphite with plastic binders are also incompatible.

(U) With fluorine, the key to using compatible materials has not been so much the choice of metals as the cleanliness. Most erratic behavior and ignition of metals has resulted from contamination. Most metals show little or no signs of corrosion following prolonged exposure to contaminant-free LF_2 . However, most metals are attacked severely by HF, particularly in the presence of free moisture (Reference 11). By choosing basically compatible materials, corrosion problems involved in handling any of the fluorine oxidizers will be relatively insignificant if HF can be excluded from the system and the oxidizer kept in a high state of purity.

(U) NASA has conducted tests with streams of LF_2 impinging on material samples at flow velocities up to 400 pfs and pressures up to 1,500 psig. No measurable physical attack occurred with nickel, stainless steel, aluminum, or brass specimens. The results of the tests showed that turbulence, fluid friction, and impact effects resulting from high-pressure, high-velocity LF_2 flow through clean tubing or past irregularly shaped or sharp-edged objects will not initiate system failures (Reference 11).

7. MANUFACTURING AND INSPECTION TECHNIQUES

(U) The primary components of the QD coupling will be made from metal by machining processes. These parts include the bodies for the two coupling halves, the interface seal, the latching mechanism, and the release mechanism. For a 2-in. diameter coupling, the bodies will probably be machined from bar stock. Other possibilities are to start with a forging billet or a casting. For the limited quantities which will be required for development hardware, it is not economically justifiable to make the tooling for forgings. The use of castings presents other problems which have not been sufficiently investigated to warrant their use. These problems include contaminant inclusions and difficulties in welding.

(U) The most critical machined areas are the sealing surfaces. They must be relatively flat, smooth, and free of imperfections such as nicks and scratches. Two concepts for metal-to-metal seals were considered. In one concept, the interface seal is formed by mating two hard metal surfaces with extremely fine surface finishes. In this case, the sealing forces cause stresses at the sealing surfaces less than the yield strength of the materials. Sealing is effected by closing the gaps between the two mating surfaces by elastic deformation of the materials until a continuous ring of contacting area is achieved. In the second concept, a soft metal seal ring is sandwiched between two hard metal surfaces. The hard surfaces are designed so that their area of contact with the soft

seal ring is initially very small (sharp corners or knife edges may be used). The sealing force must be sufficiently high to cause plastic deformation of the seal ring, thereby permitting all of the gaps between the sealing surfaces to be closed.

(U) The machining is much more critical for the hard-to-hard (elastic deformation) metal seal approach. The amplitude of waviness of the mating surfaces must not exceed a few microinches and a surface roughness in the order of 1μ in. or less is necessary if a reasonably low leakage rate is to be achieved. The usual methods for achieving these near-perfect surfaces is to use a lapping technique. The production of surfaces to this degree of flatness and smoothness requires special care and skill on the part of the machinist.

(U) Flat surfaces are the easiest and least expensive to lap because they may be produced on a flat lapping table, whereas for contoured surfaces such as spherical or conical surfaces, a contoured lap of the same size and shape as the finished surface must be made. One of the principal problems in lapping of flat surfaces is the tendency of the edges of the surfaces to round off as a result of the particles in the lapping compound piling up in front of the surface being lapped as it is moved over the lapping table.

(U) Additional problems are encountered in protecting the surface from damage after it has been machined. It must be handled carefully to keep it from being damaged by contact with hard objects such as tools, work tables, and inspection instruments. When the two sealing surfaces are being mated, they must be aligned before contacting each other and must not be permitted to slide with respect to each other because the sliding motion can cause scratches or galling which could prevent a satisfactory seal.

(U) The matching of the components for the soft metal seal is much less critical. Since the sealing loads cause the harder mating components to cut into the soft seal, a surface finish of 125 arithmetical average (AA) μ in. is adequate for the soft part. It is not permissible to have pits or scratches in the surface which are deeper than the specified maximum surface roughness. It is desirable to have the surface produced by a turning operation so that the machining grooves form a circular lay and there will be a minimum number of these grooves crossing the sealing area. The sharp corners or knife edges on the hard components must be free of nicks and the amplitude of waviness of these sealing edges measured in the direction parallel to the axis of the coupling should not exceed 250μ in. peak-to-valley. The roughness of the intersecting surfaces which form the sealing edges should not exceed $32AA\mu$ in.

(U) Although the soft metal seal version does not have as stringent requirements for surface finishes as the hard seal version, it is also vulnerable to damage from careless handling and must be given the

same considerations during handling and mating of the coupling as noted above for the hard seal version.

(U) It is expected that normal tolerances and surface finish requirements will apply to the remainder of the machining operations on the coupling and it will be capable of being produced in any machine shop with standard milling and turning machine shop with standard milling and turning machines.

(U) Inspection methods for surfaces finished to the tolerances specified above for the hard metal seal coupling must be limited to optical methods of measuring flatness and smoothness. The use of inspection equipment such as a Profilometer or a Proficorder which utilize a stylus to trace over the surface for measuring roughness are not permissible because the stylus may cut a groove deeper than the surface irregularities being measured and thereby create a leak path. The optical inspection devices which are useful in measuring the quality of a surface finish are the optical flat and the interference microscope. Both of these devices make use of the phenomenon of optical interference.

(U) Besides machining, the manufacturing processes which may be applicable to the QD are welding and brazing. Since it is desirable to control the leakage of the toxic fluorine related oxidizers to a minimum the transfer systems should contain a minimum number of mechanical joints. Welding or brazing should be used wherever possible for joining components. Typical applications for welding include joining the body of the coupling to the vehicle fill line, joining the body of the coupling to the AGE umbilical fill line, adding bosses to the body for attachment of drain and purge lines, and addition of external flanges and lugs to the body for mounting to the vehicle or for attachment of the latch mechanism. Weld joints should be carefully designed so that after welding there will be no cavities or crevices formed by layers of metal exposed to the oxidizer. Weld joints should be butt joints rather than lap joints.

(U) The following procedures for welding a pipe joint for a fluorine system is recommended in Reference 11 and is applicable for any welding required on the QD.

(U) Pipe ends to be welded should be beveled suitable for full-penetration V-notch butt welding. Sleeve joint should be avoided unless the overlap joint can be brazed or isolated by welding. Welding should be performed by a qualified welder using a shielded arc (heliarc) with an inert gas backup so that the inside of the pipe is not contaminated by slag or other contaminants which cannot be cleaned off by normal cleaning procedures. The weld should be protected by purging with argon or helium gas before welding is started. The purge may be reduced during welding to prevent blowout of the weld. Gas protective devices should be in place during all of the welding process. An appropriate filler ring should be used on the first pass. The remaining passes may be metal-arc'd to minimize distortion and carbon precipitation in susceptible alloys and to increase welding speed. The weld penetration or depth of fusion must

be to the bottom of the vee groove (excess penetration of a bead 1/16-in. high on the inside of the pipe is acceptable if the bead is smooth and well rounded).

(U) There must be no cracks, crater defects, pinholes, or slag. After the first pass, the welder should inspect the weld carefully for defects. Craters, cracks, and rough lumpy spots must be ground or chipped out before continuing the weld.

(U) The arc should not be broken at the centerline of the weld. The welding rate should be accelerated until the weld pool becomes small, then the arc should be moved off to the side of the groove before being broken. The bead, where broken, should have a tapered end. It is recommended that the work be turned to allow downhand welding wherever possible.

(U) Welded joints should be stress-relieved, if necessary by heat treating. Welded seams and connections used in fluorine/FLOX installations require X-ray inspection of all welded joints. Welds with poor penetration, flux or slag inclusions, pockets, bubbles, or surface flaking cannot be permitted. Welded connections must be cleaned and passivated before being placed in fluorine service.

(U) There are few comments to be found in the published literature concerning experience with brazing in fluorine systems. Gold-nickel and copper brazing alloys should be compatible with fluorine; however, it is expected that silver brazing would not be compatible. Douglas experience indicates that some silver braze alloys are rapidly attacked by fluorine as well as by a dilute nitric acid solution which is sometimes used in the cleaning of components. Before using any brazing alloy in a fluorine system, a brazed sample made from the actual metals to be joined should be exposed to fluorine and hydrogen fluoride to establish that the material actually is compatible. If brazing is used, the finished joint must be thoroughly inspected to make certain that there are no unfilled crevices remaining and that all traces of brazing flux have been removed.

(U) All brazed joints should be X-rayed to ensure that no hidden cavities or flux and slag inclusions are present. When defects in the brazed joint are found, the component must be rejected.

8. CONTAMINATION

a Importance of Contamination Control

(U) The importance of contamination control on components or systems used for service with any of the four fluorine-related oxidizers cannot be overemphasized. The principal areas of concern are reaction of the contaminant with the oxidizer and system malfunction as a result of the contamination. Because fluorine reacts vigorously

with most of the nonmetal materials, as well as with some of the metals, the presence of these materials as contaminants could initiate a reaction and cause a system overpressure or a burn-through of the walls of the oxidizer system. Types of system malfunctions which can be caused by particulate contamination are plugging of orifices or other small passages and interference with mechanical functions such as valve closing.

b. Types of Contamination

(U) For most fluid systems, the primary contaminants may be grouped into the following three categories:

- (1) Noncombustible contaminants (particulate matter such as metal chips or sand)
- (2) Combustible contaminants
- (3) Water.

(U) These categories are not really suitable for application to fluorine because water and many of the contaminants normally considered noncombustible are highly reactive with fluorine. Some of the less reactive contaminants such as metal chips, particles of metal oxides and metal fluorides, and some forms of carbon particles may remain in the fluorine oxidizers as solid particles for a sufficient time to cause a system malfunction.

(U) The most common forms of combustible contaminants are organic materials such as preservatives, lubricants, paint, solvents, lint, etc., which may produce fire or an explosion hazard. Although a sufficient quantity of water would constitute a fire or explosive hazard, it may also be detrimental when present in the small quantities found in the atmosphere because its reaction with fluorine produces hydrogen fluoride which promotes corrosion of the metal components of the system. Also, in the case of the cryogenic oxidizers, the water may build up as frost or ice on the outside of the system and create an explosive hazard in the event of an oxidizer leak.

c. Sources of Contamination

(U) Sources of contamination of the hardware in a fluid system can be divided into the following two groups:

- (1) Internal contamination (contaminants initially in the system or generated by the system).
- (2) External contamination (includes airborne contaminants, contaminated fluids, and contaminants introduced by negligence in handling or maintenance of the hardware)

(U) Considering a complete oxidizer transfer system which would be required in conjunction with the subject QD coupling, internal contamination may be present before the system is operated for the first time. The sources of this type of contamination are from the manufacturing, assembly, and installation operations, as well as from contaminated storage tanks and test fluids. Some types of contaminants resulting from manufacturing operations may be hard to detect and remove. If abrasive grinding or lapping materials are used, they may be left imbedded in the surface of the metal component. Also, if welds are made in areas which are inaccessible for inspection and cleaning operations, they may contain slag and scale which could cause trouble when the oxidizer is introduced. During assembly and installation, contamination may be introduced by the mechanic in the form of oil or dirt particles from his hands, as well as from his tools and work bench. Also, there is the possibility of a noncompatible lubricant being used to mate close fitting parts. During functional testing, components may be contaminated from test fixtures used as sealing closures as well as from a contaminated system supplying the test fluid.

(U) System-generated contaminants result from the wear and deterioration of components through mechanical and chemical action. Mechanically generated particles may result from the movement of surfaces with respect to each other such as the opening and closing of valves. Particles resulting from chemical action may be in the form of metal fluorides or oxides which have formed on the metal surfaces and subsequently become dislodged.

(U) External contamination may be a problem to both the interior of the system and to the exterior of the hardware if external mechanisms are used. The interior of the system may be exposed to airborne contaminants, such as sand, dust, lint, and water, at any time it is opened to mate the propellant transfer line to the vehicle, to connect the propellant transport trailer to the vehicle loading system, to connect the vent and purge system, and to replace components. Contamination may enter an opened system from the use of a temporary closure which is either contaminated or made of a material which can leave a residual contaminant in the system when the closure is removed. The fluids used in the system are also a possible source of contamination. Any solid contaminants in the oxidizers would probably not be chemically reactive but could be a source of trouble for the satisfactory operation of mechanical components such as shutoff valves. The gases used for leak checking and purging the system could carry contaminants such as water, oil, or solids which are chemically reactive with the oxidizers into the system. Contamination of the exterior of the hardware is of concern in areas where it could lead to a system malfunction such as failure of the disconnect latching mechanism to operate or failure of a shutoff valve to operate. Of primary concern in the case of the cryogenic oxidizers is the exclusion of water which not only could

cause a malfunction by the formation of ice around an operating mechanism but also could become an explosive hazard if it becomes contaminated with fluorine.

d. Effects of Contaminants

(U) The principal problems caused by contaminants in a transfer system suitable for the four oxidizers under consideration are as follows:

- (1) Interference with moving mechanisms.
- (2) Damage to sealing surfaces.
- (3) Chemical reactivity with oxidizers.
- (4) Incompatibility with filters.

(U) The types of moving mechanisms that may be found in an oxidizer transfer system include shutoff valves for the oxidizer and purge systems and the latching and retraction mechanism for the QD coupling. Close-fitting sliding surfaces are especially vulnerable to jamming by solid particles, which wedge into the clearances, thus increasing friction and, in extreme cases, causing weldment of metal surfaces.

(U) Damage to sealing surfaces of shutoff valves or of the interface seal in the QD coupling may be caused during assembly or installation by the presence of hard contaminant particles on the sealing surfaces when they are brought together. The damage may be in the form of scratches across the sealing areas or the particles may become partially imbedded in one of the surfaces and thus hold the sealing surfaces apart. For the shutoff valve, contaminant particles may damage the sealing surfaces by abrasion during flow of the oxidizer. Another possibility of damage to the sealing surfaces is that, if a contaminant particle caught between two sealing surfaces reacts chemically with the oxidizer, a leakage path larger than the initial particle size may be created as a result of a portion of the seal being consumed during the reaction.

(U) The chief concern about chemical reaction of contaminants with the oxidizers is that the reaction may have sufficient energy to cause a rupture of the system. Other lesser effects are degradation of the oxidizer and possible physical damage to some of the internal components of the system.

(U) Use of filters in particular with liquid fluorine, has been generally unsuccessful. The filter elements usually disappear after very short periods of use. It is suspected that the entrapment of contaminant particles in the filter may cause an acceleration of the corrosive effects of the fluorine on the filter element.

e. Contamination Considerations In Design (Adapted from Reference 12)

(1) Design Criteria

(U) Contamination control approaches in the design of components and systems for the four noted oxidizers may be grouped in the following categories:

- (a) Reduce the sources of contaminants in systems and components by selecting materials and mechanical designs that will reduce the rate of wear, friction, and stress.
- (b) Increase the tolerance of components to contamination by using maximum dimensional clearances compatible with functional requirements.
- (c) Protect components and systems from contaminants by means of adequate filtration (when possible) sealed modules, clean fluids, and clean environment during assembly and installation.
- (d) Provide accessibility for the inspection of systems and components for the removal of contaminants by allowing means of disassembly for cleaning, drainage, post-assembly cleaning, and maintenance operations.
- (e) Establish adequate levels of contamination control by relating the cleanliness requirements to the actual needs and nature of the system components.

(2) Component Design

(U) Use of the following in the design of components will aid in contamination control and make the component less vulnerable to malfunction as a result of minor amounts of contamination.

- (a) The interior of all fluid components should be smooth (to eliminate flaking) and continuous (to promote flushing action during flow). Pockets, dead ends, crevices, labyrinth areas, and cavities should be eliminated; they collect dirt that may be difficult or impossible to remove by cleaning operations.
- (b) Although it is desirable to limit the number of joints to minimize leakage, the design should permit disassembly where necessary for inspection and cleaning of hidden areas.
- (c) Avoid threaded joints in areas directly exposed to the oxidizer.

- (d) Strong positioning and actuating forces should be used to preclude jamming of mechanisms by particles.
- (e) Feather edges and other delicate features susceptible to cracking should be eliminated. Minimize the number of abrading surfaces and friction points. Rubbing surfaces should be carefully designed to prevent excessive wear.
- (f) Delicate design features should be protected and airborne dirt should be kept away with protective caps or covers.
- (g) The widest possible tolerances in orifices and clearances should be provided. Design the components to operate with a fluid contaminated with the largest particles tolerable.
- (h) Screw-type fasteners and other particle generating connectors or devices should be minimized in locations where the resulting particles are exposed to the oxidizer.
- (i) Dynamic mechanisms with wearable surfaces should be put through a breaking-in period to run-in the friction points and abrading surfaces. The intended working fluid should be used. After the operation, the component should be disassembled, inspected for excessive wear, recleaned, and reassembled.

(3) Materials Section

(U) Wear and corrosion of components constitute large sources of contamination; to reduce them, proper attention must be given to the process of materials selection and application. The following guidelines are recommended:

- (a) Select materials according to the following steps:
 - (1) Determine expected service conditions for the parts.
 - (2) List the materials resistant to corrosion by the oxidizer, the external environment, and combinations of both and also that fulfill the mechanical requirements of the parts.
 - (3) From the above data, narrow the number by picking only those materials that have shown low wear in similar applications.
 - (4) Follow up by actual testing.

- (b) Hoses and flexible connections in direct contact with the oxidizer must be of compatible all-metal construction. For the cryogenic oxidizers, the materials must retain good impact strength characteristics at low temperatures. For the purge system, it is permissible to use Teflon hose reinforced with stainless steel braid for the part of the system that is isolated from the oxidizer by a shutoff valve, if a filter is installed to catch any Teflon particles that flake off and prevent them from being carried into the oxidizer system during purging. Rubber hose should not be used because it is difficult to clean, and filler materials can be dislodged to contaminate the fluid.
- (c) Aluminum castings shed large quantities of very fine particles in the $3\ \mu$ range. Aluminum alloys must be anodized to prevent them from adding particles and corrosion products to the fluid stream.
- (d) Avoid the use of plating and other built-up surface finishes in direct contact with the oxidizer unless testing under simulated service conditions has demonstrated that the surface finish is completely compatible with the fluid and will not flake or peel off. The possibility of galvanic or catalytic reactions at the interface between coatings and base materials is likely; it is here that much of the apparent damage takes place.
- (e) Do not use ceramics unless they are compatible with the oxidizer and have glazed nonporous surfaces.
- (f) Soft and stringy packings are not to be used because they are gradually deposited in the fluid stream.
- (g) Development work will be required to obtain a filter suitable for use with liquid fluorine. Some of the existing filter designs may be suitable for the other three oxidizers. Before utilizing any filter in the oxidizer system, it must be thoroughly tested. Filter materials must be compatible with the oxidizer, structurally adequate, corrosion and temperature resistant, and not migrate downstream.
- (h) Careful consideration of material, hardness, and finish will reduce wear and particle generation. Surfaces that rub against each other should be smooth and of different hardness. A surface hardness of 60 RC is considered optimum for minimum wear (Reference 12, Paragraph 10.6.2.2).

(4) Manufacturing Operations

(U) The following recommendations are aimed at curtailing the generation of contaminants during manufacture of components:

- (a)** Use shrink fitting of mating parts in preference to press fitting or threading to prevent shaving off metal splinters that later contaminate the system.
- (b)** Avoid metal castings, because they generally carry inclusions and molding residues, such as core sand. Use forged or bar stock. If castings are used, the cast surfaces should be machined.
- (c)** Clean tools and molds during forming and drawing operations to avoid embedding hard contaminant particles.
- (d)** Deburr each part before forming operation, and degrease it with ultrasonic cleaning and vapor degreasing.
- (e)** Clean weld, braze, or solder joints to remove oxides. If possible, machine the weld areas exposed to the fluid.
- (f)** Use butt joints rather than lap joints for welding to eliminate areas that cannot be cleaned of contaminants.
- (g)** All functional surfaces should be finished to a level of smoothness that will permit efficient operation without generating contaminants.
- (h)** Oxides formed by heat treatment in an oxidizing atmosphere are a source of contamination. To prevent formation of oxides on surfaces to be exposed to the fluid, an inert atmosphere should be used during heat treatment. An alternative is to machine after heat treatment all surfaces that will be exposed to the fluid.
- (i)** Do not plate springs or other parts subject to high torsional stress to avoid flaking the coating.
- (j)** Avoid cored passages in castings; drilled passages are smoother and do not hold core molding materials.

(5) Component Testing

(U) To reduce possible contamination of the components during functional checkout, the following procedures are recommended:

- (a)** Maintain close control over contamination level in all test equipment. All fittings, fluids, and assemblies used to test components must be as clean as, or cleaner than, the component being tested.

- (b) Clean all test equipment connectors thoroughly before making connections.
- (c) Use dummy components to validate the cleanliness of the test circuits.

(6) Cleaning and Packaging

(U) The following practices are recommended for the cleaning and packaging of components:

- (a) Clean components and parts immediately after machining before oils set.
- (b) Clean all surfaces and channels of filters thoroughly, using a combination of ultrasonic cleaning and flushing.
- (c) Clean parts with small passages carefully to remove contaminants from these passages.
- (d) Clean at the component level, even when components are not contaminant-sensitive, to avoid contributing later to the system contamination, which is harder to eradicate.
- (e) After cleaning, package parts and components in heat-sealed plastic bags, avoiding the use of preservatives and coatings.
- (f) Cleaned components should have all ports and connections capped. Threaded male fittings should be capped with anodized aluminum caps or with clean aluminum foil. Female fittings should be plugged with the fittings used in service and capped with aluminum caps or aluminum foil. Avoid the use of plastic and soft metals for cappings. Other types of opening, such as flanged connectors or weld connect points, should be capped with clean aluminum foil secured by an adhesive tape.
- (g) A practical and feasible level of filter element cleanliness is that point when approximately the same amount of particles that could be removed by vibration and flow are removed by process cleaning.

(7) Assembly and Installation

(U) Contamination by foreign matter and airborne dust must be controlled until the component is assembled into a system. Recommendations are as follows:

- (a) Final assembly, cleaning, and inspection of components must be done in a clean environment commensurate with levels of cleanliness required.

- (b) Thread compounds and lubricants are incompatible with the oxidizers and must not be used.

9. CLEANLINESS REQUIREMENTS

a. Component Cleaning

(U) Because the fluorine related oxidizers are so highly reactive with all the organic compounds, such as oils, grease, and so on, the cleaning procedures for the components and systems used to handle these oxidizers must ensure that essentially all such reactive materials are removed prior to exposure to the oxidizer. On the basis of present experience with liquid fluorine systems, it must be assumed that filters are not compatible with the oxidizer, and therefore particles in the fluid large enough to cause a malfunction of any of the system components must be excluded by exercising the strictest control in the cleaning of components and maintaining of system cleanliness.

(U) Each user of the fluorine oxidizers develops special cleaning procedures for the oxidizer handling equipment. Cleaning methods recommended by NASA, Rocketdyne, and Bell Aircraft are presented in Reference 9. Douglas now uses a modified version of the cleaning procedures developed over a number of years for liquid oxygen components. Additional work in determining cleaning requirements for fluorine hardware is currently being accomplished by Douglas to fulfill the requirements of a NASA development program for a liquid fluorine rocket feed system. The materials and procedures presently used with complete success are as shown in the following example.

A. Cleaning Materials

A. 1 Vapor Degreasing

Solvent used for vapor degreasing cleaning processes shall be per 0-T-634, Type 2, except the nonvolatile residue shall not be greater than 0.010 gr per 500 ml.

A. 2 Component Cleaner

A. 2. 1 Trichlorotrifluorethane

This solvent, when used in the final cleaning processes, shall be per MSFC-SPEC-237. (U)

A. 2. 2 Methylene Chloride

This solvent, when used in the final cleaning processes, shall be per MIL-06998, Grade A.

A. 3 Drying or Preservation Gas

A. 3. 1 Air

Air used in the drying and preservation processes shall be prefiltered to a 100 mc level (absolute), the hydrocarbon content shall not exceed 0.5 parts per million by weight in terms of n-cetane, and the moisture content shall not exceed 26.3 ppm by weight.

A. 3. 2 Nitrogen

Nitrogen gas used in the drying and preservation processes shall be in accordance with MIL-P-27401, Type 1, and in addition, shall adhere to the purity requirements listed in Paragraph A. 3. 1.

B. Precleaning

Components, except those made of Teflon, requiring precleaning shall be processed outside the clean room and shall include removal of dirt, grit, chips, grease and other major contaminants by flushing with compressed air, scrubbing with methylene chloride using a bristle brush, and again flushing with compressed air. Repeat any or all of this procedure until the component is visually free of these major contaminants. After removal of major contaminants, the following individual material cleaning procedures shall be used.

B. 1 Low Alloy Carbon Steel

B. 1. 1 Vapor degrease using solvent per Paragraph A. 1

(U)

B. 1.2 Immerse in an alkaline steel cleaning solution. The solution concentration and temperature and the component immersion time to be in accordance with the recommendations of the manufacturer of the alkaline cleaner used. (U)

B. 1.3 Rinse thoroughly with tap water.

B. 1.4 Immerse in a solution of 10 to 15 % (by volume) sulfuric acid, 85 to 90 % water maintained at room temperature for approximately 20 min.

B. 1.5 Rinse thoroughly with tap water.

B. 1.6 Dry thoroughly with warm air.

B. 2 Stainless Steel, Monel, Inconel

B. 2.1 Vapor degrease using solvent per Paragraph A. 1

B. 2.2 Immerse in an alkaline steel cleaning solution. The solution concentration and temperature and the component immersion time to be in accordance with the recommendations of the manufacturer of the alkaline cleaner used.

B. 2.3 Rinse thoroughly with tap water.

B. 2.4 Immerse in a solution of 35 % (by volume) nitric acid, 65 % water maintained at a temperature of $125 \pm 5^\circ\text{F}$ for 20 min.

B. 2.5 Rinse thoroughly with tap water.

B. 2.6 Dry thoroughly with warm air.

NOTE: The above procedure, Paragraph B. 2, shall not be used with annealed or unhardened 440C.

B. 3 Aluminum Alloys

B. 3.1 Vapor degrease using solvent per Paragraph A. 1

B. 3.2 Immerse in an alkaline aluminum cleaning solution. The solution concentration and temperature and the component immersion time to be in accordance with the recommendations of the manufacturer of the alkaline cleaner used.

B. 3.3 Rinse thoroughly with tap water.

B. 3.4 Immerse in a solution of 15 % (by volume) nitric acid, 85 % water maintained at $125 \pm 5^\circ\text{F}$ for 20 min. (U)

B. 3. 5 Rinse thoroughly with tap water.

B. 3. 6 Dry thoroughly with warm air.

B. 4 Magnesium Alloy

B. 4. 1 Vapor degrease using solvent per Paragraph A. 1.

B. 4. 2 Immerse in a solution of 10% (by weight) chromic acid, 90% water maintained at a temperature of $140 \pm 10^{\circ}\text{F}$ for 1 to 5 min..

B. 4. 3 Rinse thoroughly with tap water.

B. 4. 4 Dry thoroughly with warm air.

B. 5 Copper and Copper Alloys

B. 5. 1 Vapor degrease using solvent per Paragraph A. 1.

B. 5. 2 Immerse in a solution of 10% (by volume) sulfuric acid, 90% water maintained at a temperature of $145 \pm 15^{\circ}\text{F}$ until the surfaces are free of oxides.

B. 5. 3 Rinse thoroughly with hot water.

B. 5. 4 Dry thoroughly with compressed air.

C. Final Cleaning

Clean all component exterior surfaces, except Teflon, by vacuuming and/or air purging, and purge the interior of the part with gas, per Paragraph A. 3. 1 or A. 3. 2, if possible prior to transferring into the clean room.

C. 1 Single Material Metallic Components and Subcomponents

C. 1. 1 Flush all significant surfaces (those surfaces that will come in contact with the system fluid) with trichlorotrifluorethane or methylene chloride per Paragraph A. 1 for 2 to 5 min.

C. 1. 2 Place in an oven at a temperature of 240 to 260°F and bake for 1 hour.

C. 1. 3 Package per Paragraph D if assembly into a component is not forthcoming within a reasonable time.

C. 2 Filters and Antivortex Devices

C. 2. 1 Disassemble the component per the applicable drawing.

C. 2. 2 Immerse parts in a component cleaner per Paragraph A. 2 for a minimum of 15 min. A nylon brush may be used to remove dirt.

C. 2. 3 Purge the interior of the filter element with a component cleaner per Paragraph A. 2.

C. 2. 4 Place in an oven at a temperature of 240° to 260°F and bake for 1 hour.

C. 2. 5 Repeat the above procedures until the component parts are visually clean.

C. 2. 6 Reassemble the components following the applicable drawing.

C. 2. 7 Package as noted in Paragraph D.

C. 3 Teflon Subcomponents

C. 3. 1 Flush all significant surfaces with a solution of water containing 1 or 2% (by volume) of detergent; that is, Alconox or equivalent, maintained at room temperature for a minimum of 5 min.

C. 3. 2 Rinse thoroughly with distilled water.

C. 3. 3 Place in an oven at a temperature of 240° to 260°F and bake for 1 hour.

C. 3. 4 Package as noted in Paragraph D if assembly into a component is not forthcoming within a reasonable time.

C. 4 Component Assembly

C. 4. 1 All necessary component assembly shall be accomplished per the applicable drawing, after individual subcomponent final cleaning has been completed.

C. 4. 2 Package as noted per Paragraph D. (U)

D. Packaging

D. 1 Small Components

Components and/or assemblies that are sufficiently small or possess external significant surfaces shall be packaged as follows:

D. 1. 1 Wrap all significant surfaces and/or openings thereto with a minimum of two layers of aluminum foil. Secure the foil with tape, completely covering all loose ends, obtaining as tight a seal as possible. Under no conditions shall the tape contact any significant surface.

D. 1. 2 Place the component in a polyethylene bag.

D. 1. 3 Purge the exterior of the bag with gas conforming to Paragraph A. 3. Exhaust the gas atmosphere from the bag by hand.

D. 1. 4 Heat seal the open bag end.

D. 1. 5 Identify as noted in Paragraph D. 3.

D. 2 Large Components

Components and/or assemblies that do not meet the limitations stated in Paragraph D. 1 shall be packaged as follows:

D. 2. 1 Place an Acliar tube over all significant surface openings. Secure the tube with tape to the external component surface, obtaining as tight a seal as possible.

D. 2. 2 Purge the interior of the component and tube with gas conforming to Paragraph A. 3. Exhaust the gas atmosphere from the tube by hand.

D. 2. 3 Heat seal the open tube end.

D. 2. 4 Identify as noted in Paragraph D. 3

D. 3 Identification

Firmly affix a tag to the outside of the packaged component without penetrating any portion of the protective bag that forms the contaminant barrier for the component. The tag shall contain the following information:

D. 3. 1 Part name.

D. 3. 2 Part number.

D. 3. 3 Manufacturer's serial number, if applicable.

D. 3. 4 Cleaned for fluorine service.

D. 3. 5 Date of cleaning.

D. 4 Packaging for Shipment

D. 4. 1 Small Components

Small parts shall be protected by wrapping with protective material and placing each part in a container prior to transporting. The parts shall be packed with the container to prevent movement. (U)

D.4.2 Large Components

Place large components in compartmented padded box, one part per compartment. Large components may also be placed on a pallet or within an individual container with designed padded covers. These components shall be secured to the pallet or within the container to prevent movement. (U)

b. Installation Requirements

(U) Care must be taken to prevent contamination of the components at the time of installation. It is frequently impossible to require that the installation of components be made in a clean room. In the case of the QD coupling under consideration, it will be necessary to install the coupling half for the vehicle in a vehicle assembly area, and the AGE half will be installed at a launch site in an outdoor environment. Observance of the following guide lines for installation of the coupling halves will limit the contamination of the components to an acceptable level:

- (1) Ascertain that the environmental atmosphere is reasonably still, dry, and free of dust. Good housekeeping practices in the indoor assembly area should keep the dust level acceptably low. For installation at the launch site, operation should be restricted to a period when wind conditions are calm and atmosphere is reasonably free of dust and dry (no rain or fog). If these conditions cannot be met and it is not feasible to wait for favorable atmospheric conditions, then a shelter with a controlled atmosphere, and suitable for making the coupling installation, should be improvised.
- (2) Components to be joined will have been cleaned for fluorine service at the component level and should remain in their sealed plastic bags until actual installation is ready to begin, thus controlling the exposure of the critical internal surfaces to the external atmosphere to a minimum time.
- (3) Visually inspect components being installed for evidence of contamination or corrosion.
- (4) Keep hands away from surfaces that will be exposed to the oxidizer to prevent contamination with oil from the skin.
- (5) Do not use a lubricant on any of the components.
- (6) Purge the interior of the system with a clean, dry inert gas after installation is complete.
- (7) Seal the open ends of the coupling with expendable closures after purging.
- (8) Cover the exterior of the coupling with a plastic bag containing a dessicant to keep dirt and moisture away from the mating surfaces and latching mechanism.

c. Mating of Coupling Halves

(U) Because the joining of coupling halves will be accomplished in an outdoor environment at the launch site, care must be exercised

to ensure that contamination of the internal areas is kept to a minimum. The following procedures should be used:

- (1) Take precautions, as necessary, to ensure that the environment is as clean and dry as practicable. The coupling should not be mated under conditions of high wind or precipitation.
- (2) Remove the temporary closures and visually inspect both halves of coupling for evidence of contamination or corrosion.
- (3) Join the coupling halves and tighten until the design load is applied to the seal.
- (4) Purge the transfer line with dry clean He, then raise the He pressure to the design pressure for the transfer system and check for leaks.
- (5) If propellant loading is scheduled within 48 hours, leave a small positive He pressure on the system until it is time to load the propellant. Then passivate the transfer system with gaseous F_2 just prior to propellant loading.
- (6) If propellant loading is not scheduled within 48 hours after the leak check is completed, close the valves that isolate the purge system from the transfer system; then cover the exterior of the coupling with a plastic bag containing a dessicant to keep out dirt and moisture.

d. Passivation

(U) Passivation of metals, as reported in Reference 9 and based on Douglas experience, with gaseous F_2 prior to exposure to liquid F_2 is not a substitute for cleaning, but is considered useful for removing traces of moisture adsorbed on metal surfaces. Passivation with gaseous F_2 at normal temperature and pressure will not remove traces of hydrocarbons from metal surfaces (Reference No. 9). Passivation should be accomplished just prior to fluorine service and the passivated surfaces must be kept in a dry inert atmosphere or the protection afforded by the fluoride film is lost. Initial passivation involves the introduction of gaseous F_2 diluted by a dry inert gas (usually N_2 or He). This is held for a few minutes, then the pressure is vented to just above atmospheric pressure. Further passivation is accomplished by increasing the F_2 concentration in steps. Final passivation should utilize pure F_2 at a temperature and pressure above contemplated operating conditions.

10. LEAK DETECTION AND MEASUREMENT

a. General

(U) The checkout, tests, and qualification of a QD coupling designed for use with fluorine oxidizers require sensitive and reliable instrumental physicochemical methods to detect concentrations, ideally, in the range of 0.1 ppm to several percent in air or purge gas.

(U) A variety of chemical reactions known to give traceable reaction products has been tested in the laboratory and has been used to detect leaks. In most cases the chemicals and sampling equipment are awkward to handle and require a highly trained engineer or chemist to use them successfully. Some development of specialized instrumentation has taken place and several types are now available. Others are in the laboratory developmental stage or have been constructed only for in-house use. The operational criteria for a satisfactory detector for use in the field are as follows:

- (1) Sensitivity -- The minimum concentration of a substance that can be detected and indicated by an analytical instrument or method. At least 0.1 ppm full scale.
- (2) Specificity for fluorine, CTF, FLOX, and Compound A -- The ability of an instrument to detect one substance without interference from impurities or other foreign substances. Less than 1% interference from other oxidizers.
- (3) Precision and accuracy -- Precision - the degree to which the analytical instruments or method repeatedly indicates the concentration of a substance. Accuracy - the measure of the difference between the indicated concentration and the true concentration, assumed or accepted on the basis of a standard independent method. $\pm 10\%$ or better in ppm range; $\pm 2\%$ or better in percent range.
- (4) Response -- (Response Time) -- The time interval required for an instrument or device to detect and measure the concentration of a substance. Automatic gas sampling rates permitting the following:
 - (a) Dynamic response -- 80% of final reading in 10 sec or better.
 - (b) Integrated Data (Total Dose) -- Direct readout in ppm-minutes or ppm-hours.
- (5) Range of concentration detected -- 0.1 ppm to 100,000 ppm full scale.
- (6) Read out
 - (a) Alarm -- Visual and sound.
 - (b) Remote -- Recording in blockhouse.

(7) Maintainability

- (a) Corrosion Resistance -- Against weather and acid gases.
- (b) Detector Life -- Maximum possible or paralleled units.
- (c) Ease of Calibration -- Internal or simple standard source.

(8) Portability -- Not over 75 lb with batteries and amplifier.

(9) Cost -- \$5,000 or less.

b. Commercially Available Instruments

(U) Four instruments are commercially available for leak detection and on-site monitoring. The salient features of these instruments are compared in Table IV.

c. Noncommercial Methods for Detection of F_2 and F_2 Compounds

(1) Methods based on direct F_2 measurement

(a) Spectrophotometric (Ultraviolet Absorption)

(U) The F_2 concentration in F_2 - O_2 mixtures has been determined by completely vaporizing a liquid sample, allowing it to expand into a special 10-cm-long spectrophotometer cell, and measuring the intensity of the broad absorption band for fluorine in a spectrophotometer (Reference 13). Because of the corrosive properties of fluorine, the cell was constructed of stainless steel and coated with Teflon, which served to minimize the absorptive effects of the metal. Sapphire windows were used because of their adequate ultraviolet transmission characteristics and resistance to attack by fluorine. A Beckman Model DK-1 recording spectrophotometer was used for the intensity measurements. The intensity of the absorption at a wavelength of 278.0μ , which is a function of the fluorine concentration, was measured. Unknown concentrations can be determined immediately by reading the intensity and comparing it with calibration curves prepared from known concentrations. The liquid fluorine mixtures were sampled with a Cosmodyne sampler and transferred to the spectrophotometer cell through a system containing a vacuum pump and manometer.

(U) Concentrations of F_2 that can be analyzed range from 0.5% to 100%. The precision and accuracy claimed for the method are 0.3% and $\pm 0.6\%$, respectively. This method is not applicable to CTF and Compound A analyses since they will not absorb at the same wavelength of the ultraviolet spectrum. This technique could possibly be adapted to in-line monitoring of F_2 concentrations.

Table IV

COMMERCIALY AVAILABLE INSTRUMENTS FOR F₂ DETECTION (U)

Manufacturer	Tracerlab	Mine Safety Appliances Company	Teledyne Systems Company	Davis Instruments
Detection Principle	Dry Krypton-85 Clathrate	Ionization Chamber Detects on Aerosol	Transducer operated on the principle of a micro fuel cell 200 L/min	Measurement of conductivity after gas sample ionizes in water 0.2 to 5L/min
Sample Flow Rate	0.5 L/min	4 to 10L min	200 L/min	0.2 - 5L min
Range	0 to 10, 0 to 30, 0 to 100 ppm full scale	0 to 5, 0-200 ppm	0 to 5, 0 to 50, 1-2000 ppm	0 to 10, 1 to 100 ppm
Sensitivity	0.1 ppm	0.05 ppm at 5-ppm range, 2 ppm at 200-ppm range	0.1 ppm	1 ppm as HF
Precision & Accuracy	±10% full scale	±2% of full scale	±2% of full scale	±2% of full scale
Selectivity	Selective to F ₂ and F ₂ compounds	Not selective	Not selective	Not selective
Interferences	Humidity, HF	Other acid gases--HF, HCl, N ₂ , O ₄ , etc.	All oxidizers	Any ionizable gas, such as NH ₃ , CO ₂ , SO ₂ , HCl, N ₂ H ₂ , etc.
Portability	Small (19 in. x 14 in. x 16 in.), compact, rugged	15 lb	Weight < 15 lb	Portable, 50 lb with batteries
Maintainability	Clathrate cell life limited	Fill reagent container and charge batteries every 24 hours	Maintenance required every 3 to 6 months	After 8 hours continuous use, batteries must be charged
Operational Period	Calibration good for 3 months with continuous operation at 5 ppm	1% change in calibration every 30 days	Calibration required every 2 to 4 weeks	Must be calibrated for specific gas; seldom need to change ion exchange resin
Warning System	Adjustable alarm set at all ranges	Alarm-- audio and visual	Audio and visual alarms	Alarms available
Effect of Overdose	Shuts off	Recovers after overdose	Has presettable high-concentration cutoff	Will saturate
Cost	\$3,000	\$1,250 to \$4,000	\$3,500 + accessories	\$3,000 to \$6,000
Remarks	AEC license for 50 mc of Kr-85 required	Remote recorder readout possible; need radioactive source license	Remote read out, probe mode, multiple sampling systems all available	Cannot determine F ₂ accurately because of F ₂ hydrolysis to HF

(U)

(U)

(b) Radiochemical Exchange by Use of Kryptonates

(U) Radioactive Krypton (Kr-85) is incorporated in the surface of solid silver iodide (AgI) and is released when F_2 reacts with the AgI (Reference 14). The proportionate amount of Kr-85 is detected by a Geiger tube.

(U) A kryptonate differs from a clathrate in that a kryptonate consists of any solid material into which Kr-85 has been incorporated, while a clathrate consists of a three-dimensional organic crystalline cage in which a second component such as the inert gas, krypton, has been trapped. The most commonly used clathrate is hydroquinone krypton 85. In this clathrate, the element or compound to be detected must oxidize the hydroquinone to release Kr-85, which in turn is radiochemically counted. It can be used to detect F_2 , O_3 , ClO_2 , and other oxidizers. One of its severest shortcomings is its sensitivity to relative humidity, which must be compensated for in calibration.

(U) In the kryptonates, the Kr-85 is entrapped in a lattice of solid. To release the Kr-85, the kryptonated solid must undergo a chemical reaction to destroy the surface layers. Presumably the kryptonates can be made to have greater specificity for detecting gases, because a solid can be used that will only react with the gas to be detected. Parametrics, Inc., have developed a kryptonate in which the F_2 reacts with AgI to form AgF and release I_2 , which in turn releases the Kr-85, which is radiochemically counted. This company has developed one instrument for use at Wright Field. It is superior to the clathrate because it is selective for F_2 and is not sensitive to relative humidity, HF, or O_3 . The kryptonated cell has a life of several weeks at the 1-ppm level but will only last 1 hour if used at the 1 to 5% F_2 level. From the weight of the kryptonate, and knowing flow rate and F_2 concentration, cell life can be calculated. FLOX, CTF and Compound A should also be detected by this instrument, because, they will react with the AgI.

(c) Electrochemical

(U) This oxidation-reduction instrument was designed and assembled at General Dynamics/Astronautics (Reference 15). The cell consists of a glass tube in which a platinum gauze electrode and a silver wire electrode are immersed in a lithium chloride solution. When an atmospheric sample is bubbled through the lithium chloride solution, any fluorine present will oxidize an equivalent amount of chloride to chlorine. The electromotive force (EMF) developed by the cell is a function of the partial pressure of chlorine and, therefore, of the partial pressure of fluorine in the

atmospheric sample. The EMF developed in the cell produces a proportional electric current in the external circuit, the value of which is continuously recorded on a strip-chart recorder. In the absence of fluorine, the cell still produces a small EMF as a result of the difference in electrochemical potential of the silver and platinum electrodes, but this is nulled by a bucking voltage provided by a small battery.

(U) The atmospheric sample is drawn through the cell at a nominal flow rate of 220 cc/min.

(U) The instrument is 14-1/2 in. wide by 10-1/2 in. high by 11 in. deep and weighs 32 lb. It is completely portable and powered by batteries. No provision is made for remote readout.

(U) Normal ranges of concentrations of fluorine determined are 0 to 5 ppm and 0 to 10 ppm. It could possibly be adapted, by buffering the electrolyte, to 1,000 ppm detection. The response is approximately 90% in 30 sec in the 0 to 5-ppm range.

(U) While the instrument is selective to fluorine, with no HF interference it should also detect CTF, FLOX and Compound A, individually, since they undergo similar reactions with lithium chloride.

(U) The cost to build the instrument was reported as \$1,200.

(d) Mercury Reaction

(U) Gaseous fluorine oxygen mixtures are introduced into an apparatus and mercury is added to react with the fluorine (Reference 16). As a result of the reaction, in which the fluorine gas is reduced by the mercury to form mercuric fluoride, the pressure in the apparatus is reduced. After completion of the reaction, Kel-F polymer oil is added to restore atmospheric pressure in the apparatus. The volume of oil plus the volume of mercury equals the volume of fluorine in the sample. The apparatus is a simple glass setup, not requiring vacuum-type stopcocks, because it is operated at near-atmospheric pressures. A manometer and vacuum manifold (to 1 mm Hg pressure) are needed. Part of the apparatus is submerged in a constant temperature bath. This technique was used to analyze fluorine in approximately 30% fluorine oxygen mixtures. A precision of $\pm 0.3\%$ was indicated from 5 tests.

(U) These operations would be limited to laboratory use and to the determination of percentage amounts of fluorine, such as in FLOX. CTF and Compound A could be analyzed in the same manner, since they will also react with mercury.

(e) Chemical Fluorine and Fluoride Dosimeter

(U) General Dynamics Corporation has also packaged this instrument, which operates as an absorber of atmospheric contamination and, as such, gives only the total integrated dose for the test run. In operation, an atmospheric sample is purged through a cylinder containing an absorber solution of 1% potassium iodide and then is expelled through the outlet tubing. The potassium iodide solution absorbs both fluoride (from any hydrogen fluoride formed from hydrolysis of fluorine) and fluorine and further reduces the absorbed fluorine to fluoride, and equivalent amount of iodide being oxidized to iodine in the process.

(U) At the conclusion of a test run, the absorber solution is removed to the laboratory and analyzed for total fluoride and iodine. The iodine is probably titrated with sodium thiosulfate. One of a number of methods could be used to determine total fluoride. The amount of iodine present is proportional to the amount of fluorine absorbed and the total fluoride present minus the fluorine absorbed is proportional to the amount of hydrogen fluoride absorbed. Alternately, the fluorine and hydrogen fluoride content could be determined as Levy and Copeland (Reference 17) did by titrating the liberated iodine with sodium thiosulfate to determine the fluorine content, and then, by adding potassium iodate solution, further iodine is formed from the hydrogen fluoride present and is then titrated with sodium thiosulfate.

(U) The packaged instrument, prepared by General Dynamics, is 14 in. wide by 12-1/2 in. high by 8-1/2 in. deep and weighs 24 lb.

(U) The nominal instrument sampling rate is 220 cc/min.

(U) Power to operate the pump is supplied by batteries.

(U) The approximate cost of the instrument is \$1,000.

(U) The range of concentration determined is 20 to 200 ppm for fluorine and 0 to 550 ppm for hydrogen fluoride.

(U) This type of detection should also determine FLOX, CTF, and Compound A since they will oxidize iodide to iodine.

(f) Gas Titration Analyzer (Continuous Monitor)

(U) An automatic fluorine analyzer was developed at Oak Ridge National Laboratory by C. W. Weber for monitoring on-stream gas systems (Reference 18). It is based upon a gas titration of fluorine with sulfur dioxide and the detection of the subsequent reduction in molar flow. A gas titration consists of transferring two gases, each from a chamber called a buret, into a reactor, where chemical combination is reduced. The volumes of all chambers, or at least the relative volumes, have been predetermined. The titration is followed by plotting the pressure changes in the reactor after each incremental addition of the titrant gas. The method is applicable to most gaseous reaction, that bring about a change in molar volumes. The change in molar flow is apparent in the reaction

$$\text{SO}_2 (\text{g}) + \text{F}_2 (\text{g}) \rightarrow \text{SO}_2 \text{F}_2 (\text{g}).$$

(U) The greater the fluorine content of the sample gas, the greater the flow change. Adapting the gas titration to a continuous analyzer involved the continuous introduction of the reactants and removal of the products, proper reaction conditions, and appropriate means of detecting the molar flow changes, which indicate the concentration of the unknown. This was accomplished by use of metering capillaries, pressure control valves, and pressure transmitters, which converted the gas pressure signals to air pressure signals, various pressure gages, and so on. The resultant gas flow product, which varies with the fluorine concentration, was reflected in the forepressure-signal changes at an exit constriction. The exhaust gases from the analyzer were removed and disposed of with chemical traps and a mechanical vacuum pump. With the reactor maintained at 200°C, reaction was complete for 0.1 to 91% fluorine. The residence time in the reactor was 2.5 to 5.0 sec. The flow rate for sample and reagent was 25 standard cc min. Seventy samples analyzed in the range from 5 to 60% fluorine matched a calibration curve within $\pm 2.5\%$ fluorine at the 95% confidence level. Precision is $\pm 1\%$ fluorine. Neither oxygen nor hydrogen fluoride interfere; therefore, it would be possible to analyze FLOX by this method. Materials similar to CTF and Compound A were not tested but UF_6 did not react.

(g) Thin Film Sensor

(U) This method is based on the detection of toxic vapors by their reaction with thin metal films to reduce the mass of the metal. The rate of metal removal is dependent on the concentration of the vapor. Because the electrical resistance of a conductor is inversely dependent on its cross-sectional area, a reduction in metal mass results in

a reduction of cross-sectional area and thus an increase in electrical resistance. The amount and rate of reaction of the thin film under controlled conditions, therefore, is proportional to the amount and rate of change of electrical resistance and can be measured with proper instrumentation.

(U) Thin film sensors for the detection of low concentrations of nitrogen tetroxide and fluorine were developed by the Magna Corporation (Reference 19). They found that silver films deposited on plastic substrates and sensitized with potassium chloride had the highest sensitivity for both nitrogen tetroxide and fluorine. The effects of temperature and humidity were studied and found to be significant in some cases, but not large enough to prevent use of the sensors over a wide range of atmospheric conditions. The sensors can be used to provide an indication of exposure to toxic vapors of nitrogen tetroxide and fluorine. A portable instrument was devised that could be carried in a pocket or worn on a belt. The instrument will respond to nitrogen tetroxide in concentrations of 0.05 ppm and to fluorine in concentrations of 1 ppm, in air, within $\pm 30\%$ of the true value. The effects of other toxic vapors were not studied. The instruments were developed as a study for the Air Force, hence the company has not pursued further development or manufacture.

(2) Methods Based on Replacement of Fluorine with Chlorine and Its Subsequent Detection

(U) Fluorine quantitatively displaces an equivalent amount of chlorine by reaction with sodium chloride and, in turn, the quantity of liberated chlorine is determined by various methods, which could not readily be applied to fluorine directly.

(a) Thermoconductivity Measurement

(U) This technique has been applied to analysis of fluorine in fluorine-oxygen mixtures, where the measurement of fluorine is too similar to oxygen to be detected but where chloride can be readily detected (Reference 20). These detectors work on the principle that gases differ in their heat conduction properties. The sample gas is passed over a heated wire in an analysis cell. The resulting change of temperatures of the wire will change its resistance. This results in unbalancing a bridge circuit that has been calibrated in terms of concentration of the gas to be detected. While the detector is portable, the sodium chloride reactor may restrict the method to the laboratory. Concentrations of fluorine from 0 to 20% in oxygen were analyzed by this method. It should be applicable to the determination of CTF and Compound A, because these also will displace chlorine from sodium chloride.

(b) Photometric

(U) The concentration of chlorine, liberated from the reaction of fluorine with sodium chloride, is measured by passing the gas through a chlorine-sensitive colorimeter, which generates an electrical signal directly related to the concentration of fluorine in the samples (Reference 21). The analyzer consists of the heated sodium chloride reactor, flow colorimeter, pressure-control system, flow limiter, a vacuum pump for drawing a continuous sample through the analyzer, and a recorder. The colorimeter used was a Beckman Model 370C flow colorimeter, fitted with spectral filters to measure the chlorine absorption at the wavelength of 360μ . A 10-cm-long nickel cell with fluorothene windows was used for the optical cell. The range for the instrument with the 10 cm cell length and 500 mm of Hg pressure was 0.05 to 15% fluorine. With a shorter cell or a lower pressure, up to 100% fluorine could be determined.

(U) Hydrogen fluoride has no effect on the analyzer, but while bromine and certain oxides of nitrogen cause optical interferences. Ozone would probably interfere by liberating chlorine. CTF, Compound A, and FLOX could be determined by the analyzer because they also liberate chlorine.

(U) The analyzer requires intermittent renewal of the sodium chloride, the chemical trap, and the recorder chart, occasional replacement of the tungsten source lamp in the colorimeter, and cleaning of the colorimeter cell as indicated by inspection.

(U) The relative standard deviation of the analyzer is about $\pm 1.5\%$ above 0.8% fluorine.

(3) Methods

(a) Gas Chromatography

(U) A gas chromatograph utilizing only Monel and stainless steel parts was designed at Rocketdyne for analyzing fluorinated materials (Reference 22). A gas chromatographic substrate was developed from 50% Kel-F polymer plasticized with 50% Halocarbon oil. This substrate is classified as a monophase gel and is vastly superior to the duophase columns for separating fluorinated materials. A hot-wire thermoconductivity detector, with the hot wires coated with Teflon, was used. Rocketdyne claims analyses of fluorine, CTF, and HF are possible, and from its data it appears that the compounds could be determined even if present in mixtures. Small gas samples were used for analysis, and no attempt was made to use the instrument for in-stream analysis. Gas liquid chromatography was applied to in-line

analyses of plant streams containing CTF, Cl_2 , HF, and other reactive gases at the United Kingdom Atomic Energy Installation at Capenhurst, England (Reference 23). A split-column chromatograph was developed and automated.

(U) Clemons and Altshuller (Reference 24) used a Micro-Tek dual-column gas chromatograph, equipped with an electron-capture detector mounted in parallel with a flame ionization detector, for studying various halogenated compounds. It is interesting to note that they made sample dilutions of 250,000,000 to 1 with nitrogen.

(b) Radioactive Cl-36

(U) This technique is being developed at Douglas. Radioactive sodium chloride will be prepared from Cl-35 . The fluorine to be determined will be passed over the sodium chloride to release radioactive Cl-36 . This chlorine, equivalent to the amount of fluorine, will be detected by radiochemical counting techniques.

(c) Infrared absorption

(U) Infrared absorption might be used for detecting CTF and Compound A directly or by their reaction with carbon to form CF_4 , which, in turn, could be detected by infrared absorption. The conversion to CF_4 could be used for fluorine and FLOX detection. Detectors have been developed using infrared absorption for N_2O_4 and UDMH.

(d) Microwave Detectors

(U) Microwave (1 mm to 30-cm wavelength) detectors are being developed where positive identification of specific compounds is possible.

(U) The interaction of a unique microwave frequency with a molecule of vapor will result in its partial absorption by the molecule. This energy can be detected and measured by electronic methods.

d. Summary

(U) None of the instruments available commercially or those known to be for laboratory test satisfactorily fulfill all the requirements for leak test measurement and site monitoring.

(U) A new instrument or an adaptation of a currently available instrument should be used to detect and measure leaks in the QD coupling for the following:

- (1) Internally -- From the vehicle or AGE shutoff valves following transfer and purging.
- (2) Externally -- From the sealing surfaces into the volume surrounded by the frost-prevention shroud.

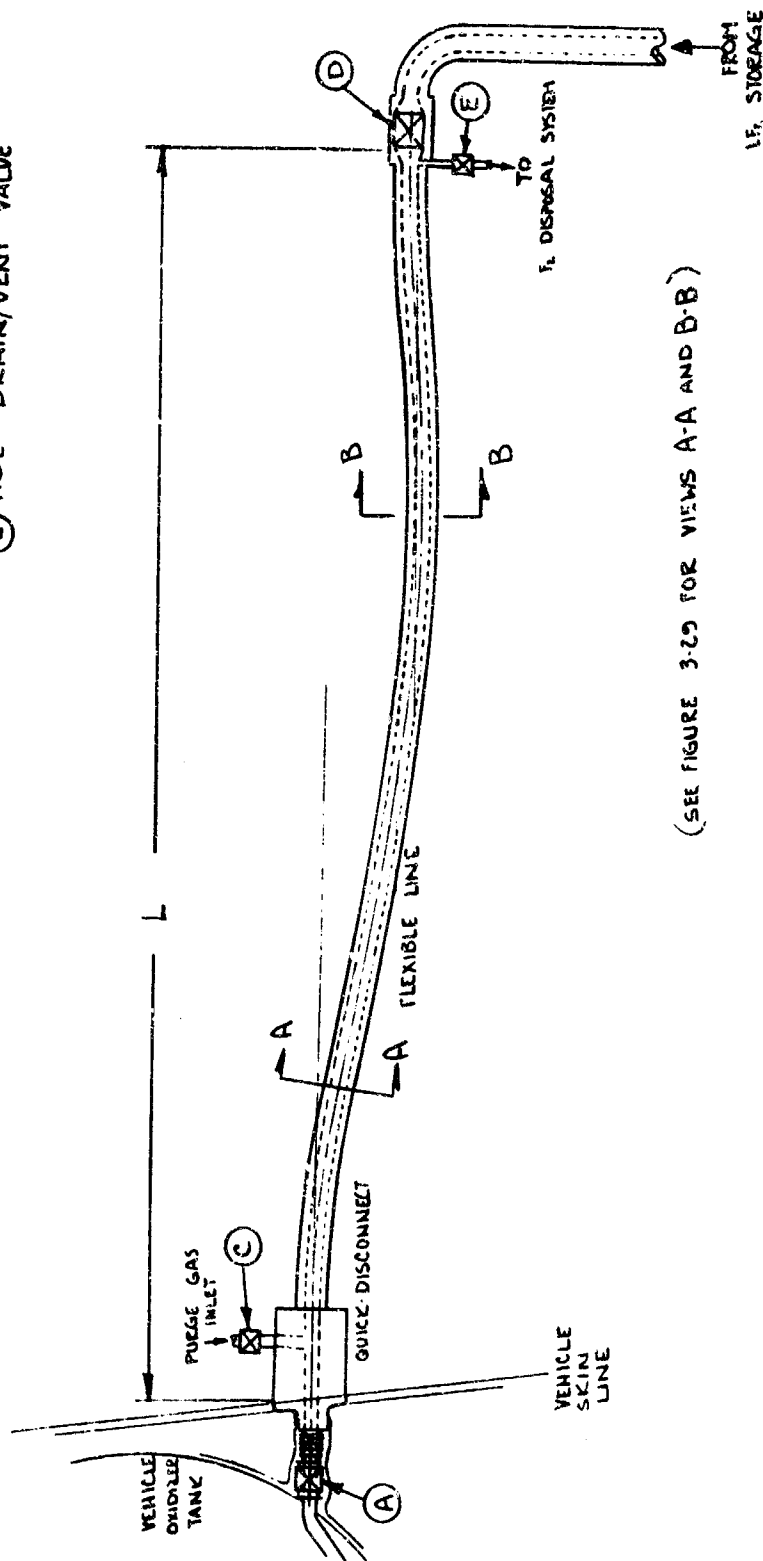
11. ANALYSIS OF LIQUID FLUORINE VAPORIZATION AND REMOVAL METHODS

a. General

(U) The oxidizer transfer lines should be arranged to promote complete gravity drainage, but irregularities and trap points in the flow channel will undoubtedly exist in a real system. A portion of the transfer line between the vehicle and AGE must be flexible to ensure proper retraction from the vehicle after separation and to prevent excessive loads on the disconnect coupling. Although they are difficult to clean properly and do not present a smooth flow channel, convoluted metal flexible lines are presently the best method to provide the required flexibility. These lines should be kept to minimum length and should be supported to minimize the trapped volume resulting from sag or slack in the line. A typical installation, such as that shown in Figure 28, may trap as much as 20% of the line volume. A well supported or positioned line (on a slope, and so forth) as shown in Figure 29, would be more likely to trap only the contents of the bellows convolutions -- 5% of line volume. Cryogenic propellants will be loaded in the subcooled state through liquid nitrogen jacketed or vacuum-jacketed lines. The heat required for vaporization of trapped liquid, then, must be introduced by design. The system may be purged internally with warm gas to vaporize liquid and remove the resulting fluids.

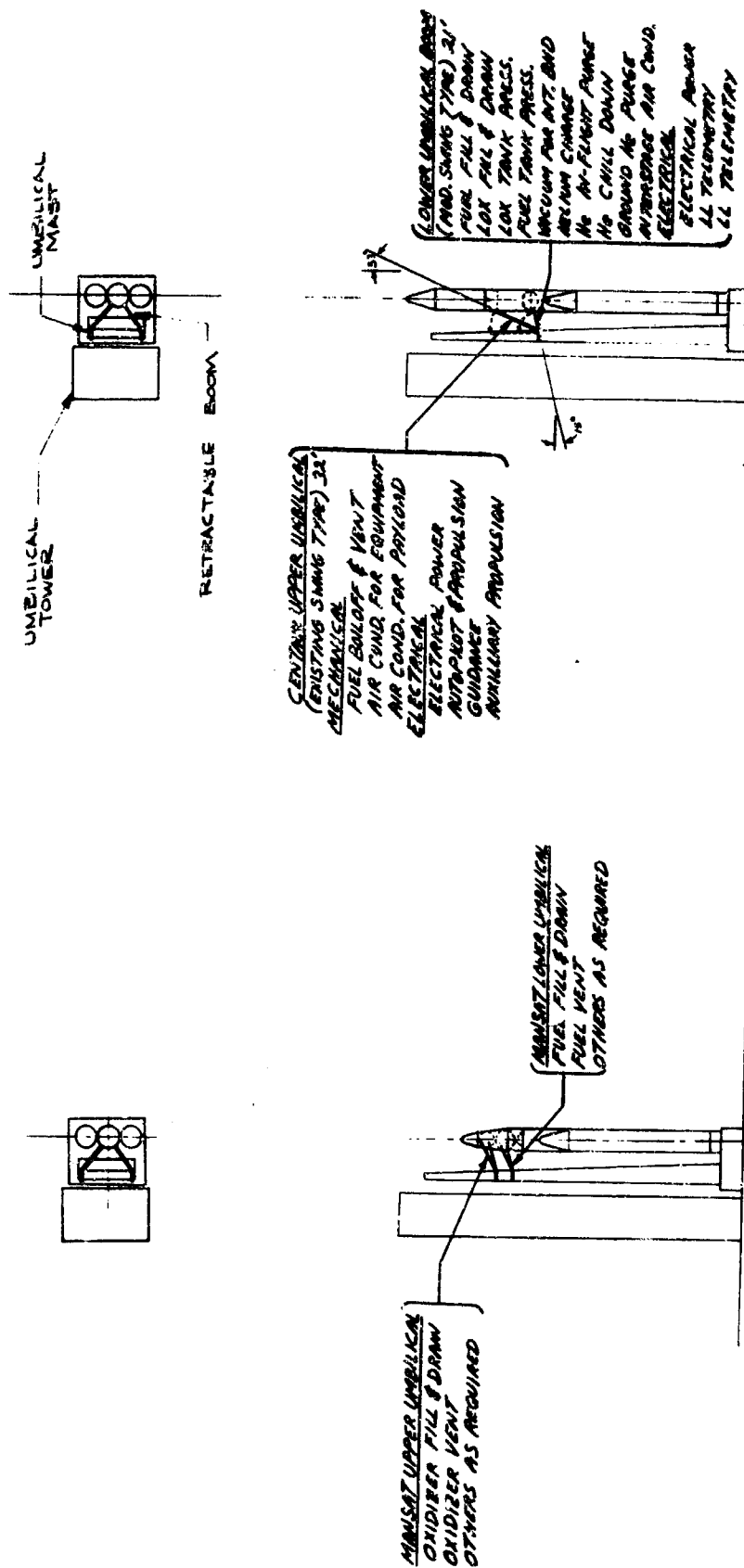
(U) The portion of fill and drain line from vehicle/AGE interface to the vehicle fill and drain valve will probably be very short and slightly elevated to promote liquid drainage. Residual liquid can be removed by directing warm purge gas into the dead-ended line to induce turbulence and ensure liquid vaporization. Turbulent flow past all surfaces of the tube can be expected if the column of gas to be agitated is of a length-to-diameter ratio less than 6. Should the vehicle design be such that length-to-diameter ratio for this portion is greater than 6, the warm purge gas must be introduced at the vehicle fill-and-drain valve end of the line.

- (A) VEHICLE FILL/DRAIN VALVE
- (C) PURGE VALVE
- (D) AGE SHUTOFF VALVE
- (E) AGE DRAIN/VENT VALVE



(SEE FIGURE 3-25 FOR VIEWS A-A AND B-B)

Figure 28. Typical Fill and Drain Line Installation



TITAN III C / MANSAT
LAUNCH POSITION

TITAN III C / CENTAUR
LAUNCH POSITION

Figure 29. Launch Positions

b. Purge System Description

(U) The purpose of this section is to interrelate the fluid flow and heat transfer parameters and develop methods of predicting flow rates and time necessary to ensure removal of the oxidizer from the trapped portions of the fill and drain line located between the vehicle and AGE shutoff valves. For this purpose, an example based on the transfer line shown in Figure 28 is chosen.

(U) Following liquid fluorine transfer, the line is gravity drained through Valve E, but some liquid remains as a result of convolutions in the flex line and other trap points in the line. It is estimated that 5% of the total line volume is trapped in the convolutes (Figure 30a), and an additional 15% of the total line volume is trapped at Section BB (Figure 30b) as a result of line sag.

(U) Nitrogen purge gas at 38 psia and 80°F is introduced through Valve C (Figure 28) and directed into the vehicle fill and drain line through directional nozzles. The gas is allowed to exhaust through Valve E to a vent and disposal system at a static pressure of 15.7 psia.

(U) Sonic flow through Valve E will result when the internal pressure reaches

$$P_i = \frac{P_v}{\left(\frac{2}{\gamma + 1}\right)^{\frac{\gamma}{\gamma - 1}}} \quad (10)$$
$$P_i = \frac{15.7}{0.5283} = 29.8 \text{ psia}$$

where

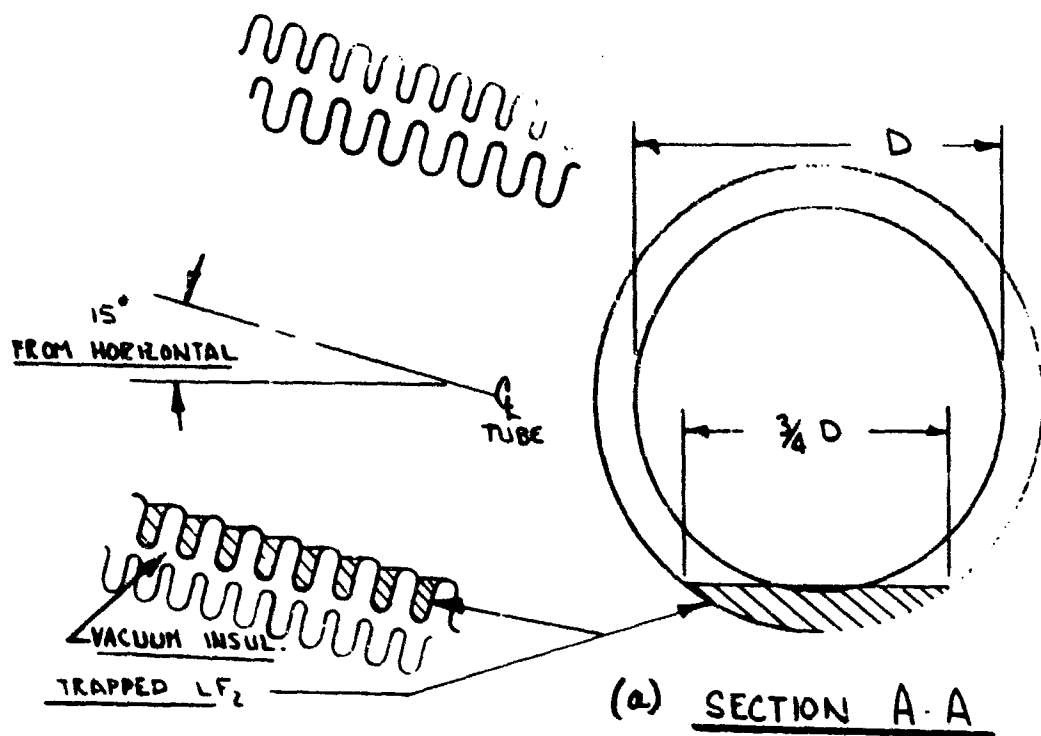
P_v = vent system pressure = 15.7 psia

and

γ = ratio of specific heats for nitrogen = 1.4.

Weight flow rate, \dot{W}_E , through this orifice at sonic velocity is

$$\dot{W}_E = C_D A_o P_i \sqrt{\frac{g\gamma}{RT_i} \left(\frac{2}{\gamma + 1}\right)^{\frac{\gamma + 1}{\gamma - 1}}} \quad (11)$$



LF_2 TRAPPED DUE
TO SAG OR SLACK
IN LINE

(b) SECTION B-B

Figure 30. Fluorine Trap Points

where

\dot{W}_E = weight flow rate (lb/sec),

A_o = orifice area, ft^2

R = gas constant (lb-ft/lb $^\circ$ R),

T_i = 540 $^\circ$ R,

and

C_D = orifice discharge coefficient = 0.8, for a short tube

then

$\dot{W}_E = 77.1 A_o$, lb/sec

(U) When steady-state purge-gas flow has been established, the subcritical weight flow rate through Valve C is approximately equal to the weight flow rate through Valve E.

$$\dot{W}_C = C_D A_{oC} w_o \left(\frac{P_i}{P_o} \right)^{\frac{1}{\gamma}} \sqrt{2g \frac{\gamma}{\gamma - 1} R T_o \left[1 - \left(\frac{P_i}{P_o} \right)^{\frac{\gamma - 1}{\gamma}} \right]} \quad (12)$$

where

A_{oC} = orifice area at C (ft^2),

\dot{W}_C = weight flow rate (lb/sec),

w_o = density of purge gas upstream of C (lb/ ft^3),

P_o = pressure of purge gas upstream of C = 38 psia,

T_o = 540 $^\circ$ R,

then

$\dot{W}_C = 82 A_{oC}$ (lb/sec),

because

$\dot{W}_C = \dot{W}_E$

$A_{oC} = 0.94 A_o$

(U) The heat required to vaporize trapped liquid may be added by passing warm purge gas through the transfer line. Area-of-surface contact between warm gas and liquid depends on the local geometry of the tube, but is assumed to average 3/4 diameter-to-length ratio over the total tube length.

$$A_s = \frac{3/4 D L}{12} \quad (13)$$

where

D = transfer line diameter = 2 in.,

and

L = transfer line length (ft),

then

$$A_s = 0.125 L \text{ (ft}^2\text{)}$$

(U) Additional heat will be transferred to the tube walls, but because of the thin section of the tube wall the rate of heat transfer to the liquid is small. It is, therefore, conservative to neglect heat transferred to the tube walls.

(U) The rate of heat transfer to the trapped liquid is

$$q = h A_s T$$

$$q = \frac{0.125 L h T}{3600} = 3.475 L h T \times 10^{-5} \text{ Btu/sec} \quad (14)$$

where

T is the difference in temperature between the GN_2 and the trapped liquid.

h = the film coefficient of heat transfer

$$h = 0.023 \frac{k}{D} (\text{Re})^{0.8} (\text{Pr})^{0.4}, \text{ Btu/hr ft } ^\circ\text{F}$$

where

k = thermal conductivity of GN_2 = 0.015 Btu/hr ft $^\circ\text{F}$

Pr = Prandtl number = 0.7 (approx for GN_2)

Re = Reynolds number = $\frac{D w v}{\mu}$

D = tube diameter (ft),

since

$$\dot{W} = A v w$$

v = purge gas velocity

μ = viscosity of nitrogen = 12×10^{-6} lb/sec ft

w = specific weight for GN_2

then

$$v = \frac{\dot{W}}{A w} = 286 \dot{W} \text{ ft/sec}$$

and

$$\text{Re} = 6.4 \dot{W} \times 10^5$$

(U) The GN_2 temperature drop (ΔT) due to LF_2 vaporization is

$$\Delta T = \frac{q}{\dot{W} C_p} \quad (15)$$

where

C_p = specific heat, constant pressure = 0.25 Btu/lb $^{\circ}\text{F}$.

Now the time required to vaporize all the trapped LF_2 can be determined as follows:

$$t = \frac{V \lambda w}{q} \quad (16)$$

where

t = time (sec)

λ = heat of vaporization for LF_2 = 74.1 Btu/lb,

V = initial volume of trapped liquid, ft^3 ,

and

w = density of LF_2 = 94.3 lb/ ft^3 ,

then

$$t = \frac{2V}{Lh \Delta T} \times 10^8$$

(U) Figure 31 represents time required to vaporize 20% trapped liquid from a 20-ft transfer line as GN_2 purge gas flow rate is increased. Figures 32, 33, and 34 represent the variation of time required to vaporize all LN_2 with line length for several GN_2 flow rates.

c. Residual liquid in the Vehicle Fill and Drain Line

(U) For simplicity in the purge system, the GN_2 should be introduced through the ground half of the QD coupling. If the vehicle portion of the fill and drain line is short ($L/D < 6$), residual liquid trapped in convolutions can be removed by introducing purge gas through nozzles pointing into the tube at a 45° angle to the axis, as shown in Figure 35. Because the gas must flow inward near the walls and outward near the center of the tube, a conservative estimate of velocity at the wall is made by assuming expansion from the nozzles to half the area of the tube and that velocity diminishes linearly to zero at the closed end. Because the nozzles are directed at a 45° angle to the tube axis

$$v = \text{initial velocity at wall} = \sin 45^\circ \left(\frac{\dot{W}RT}{PA} \right) \text{ ft/sec} \quad (17)$$

$$v_2 = \text{velocity at closed end} = 0 \text{ ft/sec}$$

If

$$\dot{W} = 0.15 \text{ lb/sec}$$

$$R = 55.3 \text{ ft/}^\circ\text{R}$$

$$P = 38 \text{ psia}$$

$$T = 530^\circ\text{R}$$

$$A = \frac{1}{2} (2)^2 \frac{\pi}{4} = 1.57 \text{ in.}^2$$

then

$$\bar{v} = \text{average velocity at wall} = \frac{v_1 + v_2}{2} = 26 \text{ ft/sec}$$

The Reynolds number is

$$\text{Re} = \frac{D w v}{\mu}$$

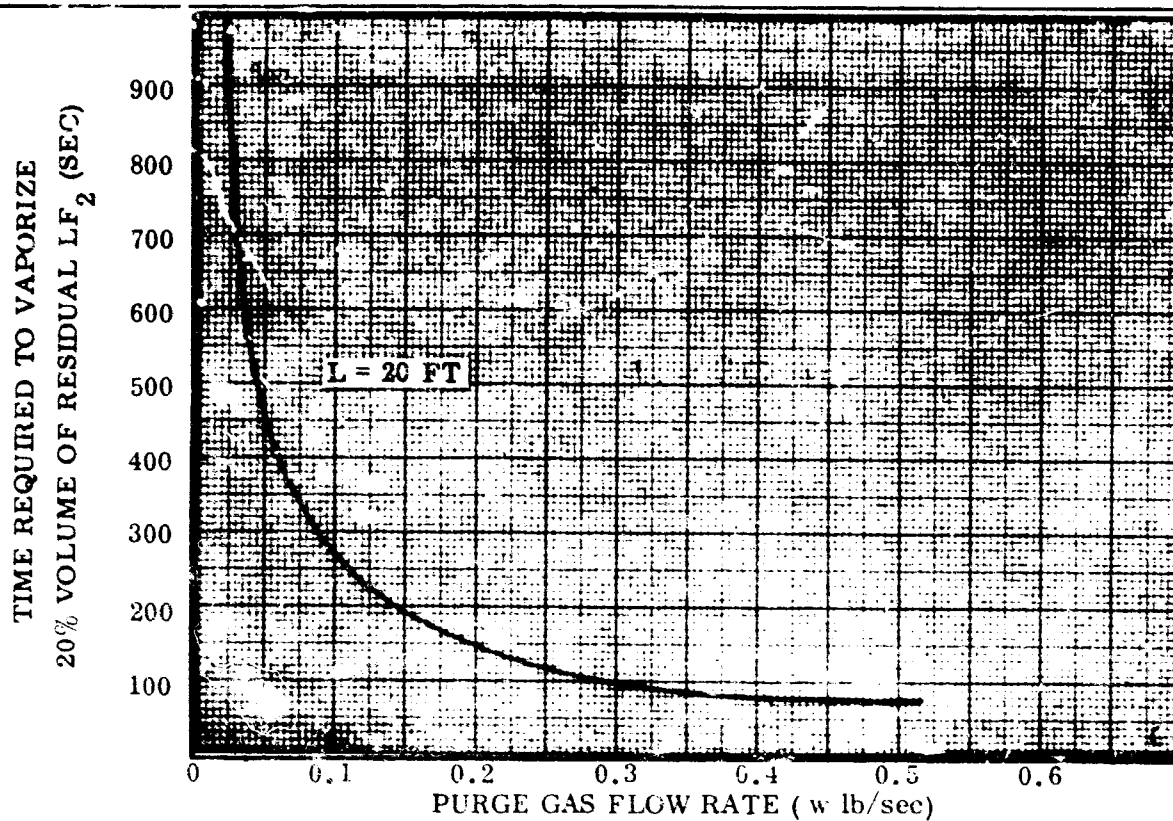


Figure 31. LF_2 Vaporization Versus Purge Rate

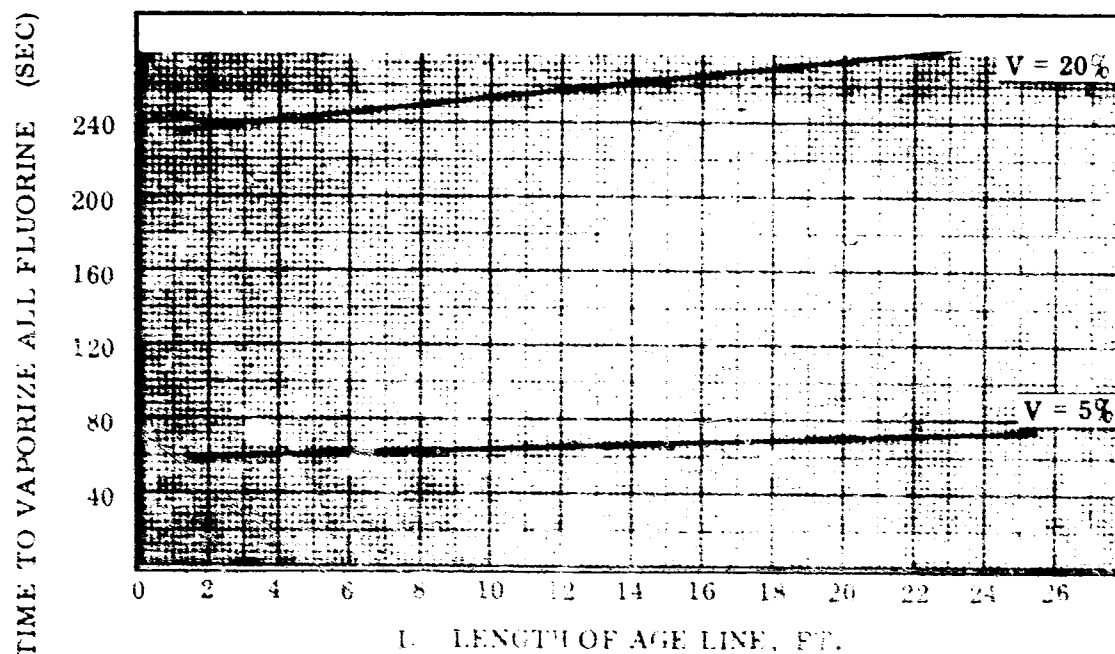


Figure 32. LF_2 Vaporization for 0.10 lb. sec Purge Rate

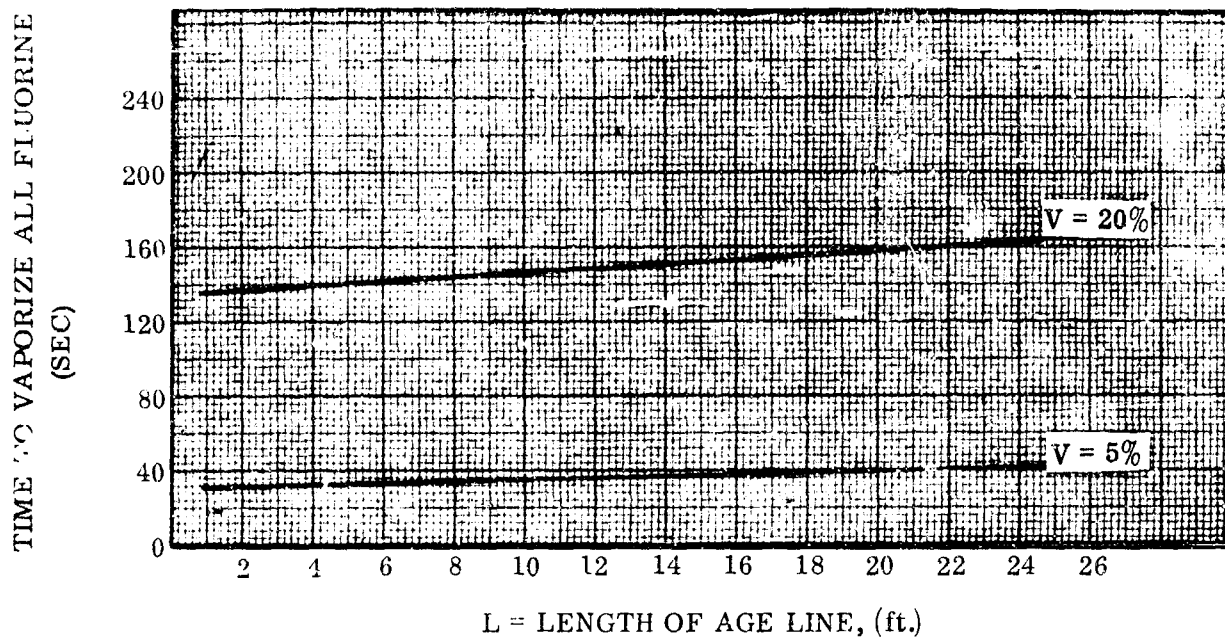


Figure 33. LF_2 Vaporization for 0.20 lb/sec Purge Rate

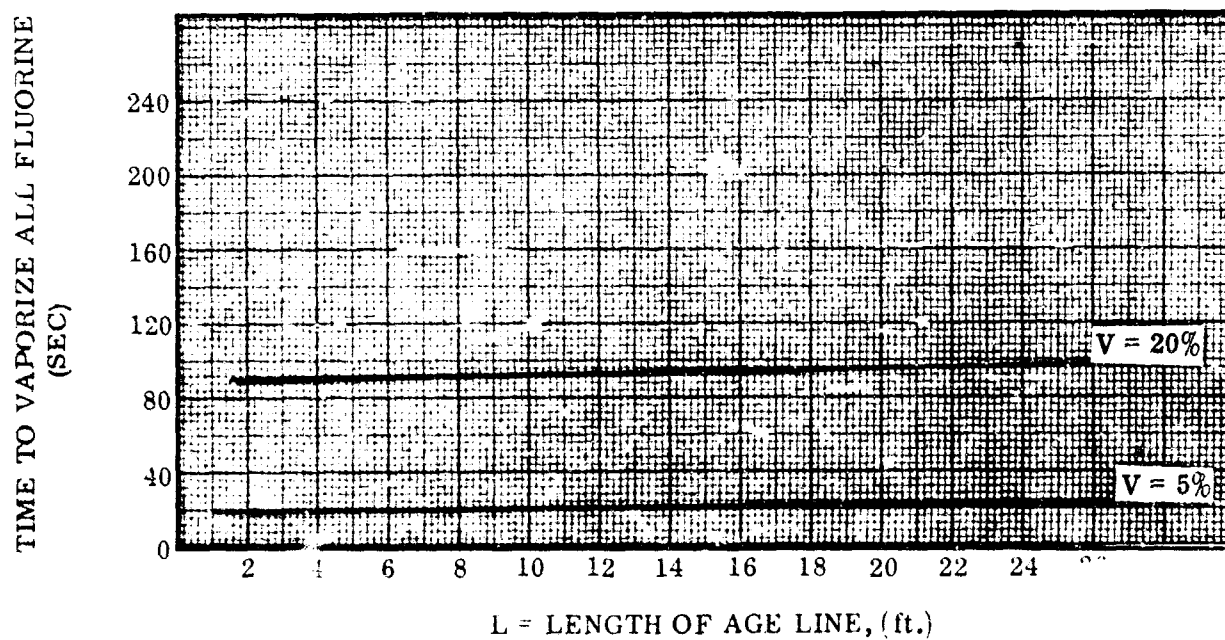


Figure 34. LF_2 Vaporization for 0.40 lb/sec Purge Rate

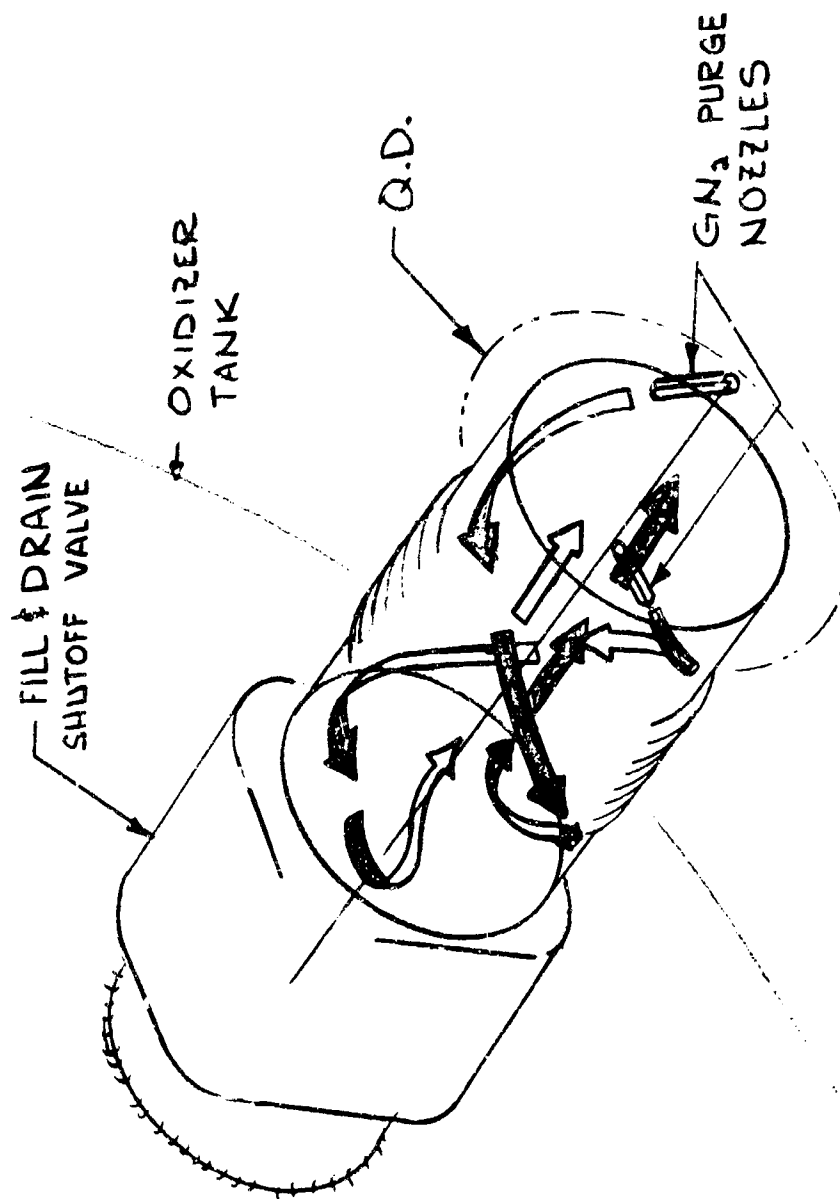


Figure 35. Forced Circulation of Purge Gas in a Short Vehicle Fill and Drain Line

where

$$D = 1/6 \text{ ft}$$

$$w = \text{specific weight} = 0.14 \text{ lb/ft}^3$$

$$\bar{v} = 26 \text{ ft/sec}$$

$$\mu = 12 \times 10^{-6} \text{ lb/sec ft}$$

then

$$Re = 5.08 \times 10^4$$

and the film coef. for heat transfer at the wall is

$$h = 0.023 \left(\frac{k}{D} \right) (Re)^{0.8} (Pr)^{0.4} = 11.3 \text{ Btu/ft}^2 \text{ min}^\circ \text{F} \quad (18)$$

(U) If it is assumed that residual liquid fills 5% of the tube volume and that liquid surface area is 3/4 diameter times length of the tube, then LF_2 vaporization rate per foot of tube length is

$$\frac{q}{74.1 \text{ ft}} = \frac{h A_s \Delta T}{3600 \times 74.1 \times \text{ft}} = 0.00185 \frac{\text{lb}}{\text{sec}}/\text{ft} \quad (19)$$

Residual LF_2 per foot of tube is

$$94.3 \times 0.05 \times \left(\frac{1}{6} \right)^2 \frac{\pi}{4} = 0.103 \text{ lb/ft} \quad (20)$$

(U) the calculated time required to vaporize a residual 5% LF_2 from the short dead-ended tube is less than 1 min. for a purge flow rate of 0.15 lb/sec.

Section III

FAILURE MODE ANALYSIS

1. GENERAL

(U) Failure of the QD could result in the release of oxidizer in quantities that may vary from 100% of the liquid material in a brief instant of time to a slow passage of gas through minute openings or leaks in sealing surfaces or metal imperfections. The problems which can arise upon development of oxidizer escape will, of course, depend upon the rate of escape, and their severity is probably some exponential function of this rate. Another more serious factor that must be considered is the possibility of simultaneous escape of fuel and oxidizer.

(U) Another facet of the leakage problem that must be subjected to careful analysis in fluorine oxidizer systems is that of passage inward of unwanted contamination from the outside of the system. Any perforation or leak can allow passage of materials in either direction -- during periods of relatively low internal pressure (subcooled LF_2), there is the possibility of materials passing inward, while, if the system is held at close to-atmospheric pressure, repeated cycles of inboard-outboard materials transfer (breathing) become of consequence.

(U) Consider a typical cycle of vehicle fill and drain operations as shown in Table V. The possible modes of inadvertent escape of oxidizer or entry of contaminants for each operation are listed in the right-hand column. For purposes of this argument, it is assumed that properly operational, cleaned, and protected quick-disconnect couplings have been installed on the vehicle before the start of the operations listed in Table V.

Table V
INADVERTENT OXIDIZER RELEASE VS
VARIOUS OPERATING MODES (U)

Operation	Propellant Escape or Contamination Sources
Connection of vehicle to umbilical	No propellant is present. Aerosol contamination during time connector elements are outside of wrappers and before connection is made. Degree of contamination is small because of selection of proper weather conditions and purges on elements. (U)

Table V. Continued (U)

Operation	Propellant Escape or Contamination Sources
Leak check of Connection	
1. Pressure	No propellant present. No inboard contamination because of excess pressure during leak check.
2. Vacuum	Some inboard contamination possible because of vacuum in system. Serious contamination unlikely because (1) previous pressure check tentatively OK'd system, (2) large leak would indicate failure of connector (or lines).
Cryogenic check of connection	No propellant is present. Inboard leak possible if thermal contraction opens up seals, but should not happen if previous design and testing is adequately done. Severe contamination would arise from inboard leak because of condensation or frost.
Passivation with GF_2	<p>Propellant present, fairly low pressure (above atmospheric), ambient temperature. Outboard escape of oxidizer in connector area should be small enough to be handled by normal GN_2 purge of area. Concentration should be monitored to determine rate of escape and size of escape path. Increase of rate with time would be dangerous and would require correction. Connector should be enclosed to prevent access of escaping F_2 vapors onto other structural (vehicle or AGE) systems to prevent corrosion because of HF from $F_2 + H_2O$ (air) $\rightarrow 2HF + 1/2 O_2$. Inboard contamination unlikely because of excess pressure inside the system.</p> <p>Complete failure of connector or seal would release a cloud of F_2 or $F_2 - N_2$ gas (depending on time of occurrence). This could be of considerable volume during passivation.</p> <p>Partial failure of connector or seal could result in impingement of jet of GF_2 onto inside of connector enclosure and then out purge exhaust. The automatic controls on the valves (above) might not respond rapidly, and considerable F_2 may pass out purge exhaust.</p> <p style="text-align: right;">(U)</p>

Table V. Continued (U)

Operation	Propellant Escape or Contamination Sources
Purge removal of passivation gas	<p>Situation similar to passivation except that concentration of F_2 is continually decreasing. Contamination may be introduced by purge gas. No inboard leaks are assumed.</p>
Static hold of empty passivated vehicle	<p>Only traces of propellant present. Two conditions are possible: (1) lines at atmospheric pressure -- breathing through seals and leaks introduces moisture and causes deterioration of passive film and galvanic corrosion; (2) lines held at positive pressure -- any leak is outward and is not a problem. Pressure monitoring will detect leakage.</p>
Vehicle fill	<p>The period when LF_2 is being pumped through the fill line and connectors is obviously the most critical time in relation to inadvertent escape of oxidizer. If the connector is designed with a venturi present, inward leaks of contaminant may also develop.</p> <p>The most serious single incident which might occur would be a parting of the line or connector. The most serious time for this to happen would be during the period when LF_2 is flowing at its maximum rate into the vehicle tank. Simultaneous failures that would increase the problem would include:</p> <ol style="list-style-type: none"> 1. Failure of the vehicle fill and drain valve to close if the fill coupling parted, so that all oxidizers pumped into the vehicle could be discharged to the atmosphere. 2. Failure of the AGE shutoff valve, so that LF_2 continues to be pumped from a separated coupling until backup valving could be activated. <p>At the connector that is enclosed in a purge box, a jet of escaping LF_2 would impinge the inside of the box, evaporate rapidly, and be swept out the purge vent. F_2 sensors located in the vent would announce the existence of the leak and shut off the flow of LF_2.</p> <p style="text-align: right;">(U)</p>

Table V. Continued (U)

Operation	Propellant Escape or Contamination Sources
	<p>Smaller openings could be expected to allow only GF₂ to escape, again in the connector area to be swept away in the purge gas stream.</p> <p>Leaks developing downstream of the connector, i. e., inside the skin of the vehicle, would pose problems. It is unlikely that the vehicle portion of the fill and drain plumbing will be vacuum jacketed, but is more likely to be covered by multilayer (super) insulation. It has been shown that Dimplar and NRC-2 are not easily ignited by static LF₂, GF₂, or by low-pressure GF₂ jets although they do show impact sensitivity when immersed in LF₂. In general, it would be expected that other fluorine-incompatible materials would be present inside the skin of the vehicle and that a major leak would probably result in an extremely hazardous situation. The effects of LF₂ emissions would probably depend on the material upon which they impinged. Evaporation would be rapid, and the internal inert gas purge necessary to maintain the integrity of the system would be extremely large, if not impractical.</p> <p>The cycle of conditions to which the connector will be subjected during a fill operation will be as follows:</p> <ol style="list-style-type: none"> 1. GF₂, ambient temperature, volume flow rate about 10³ greater than that of the LF₂. This GF₂ is boiloff resulting from cooldown of hot plumbing. 2. GF₂, temperature decreasing steadily toward the boiling point of LF₂ at the pressure at which it is being transferred. 3. Mixture of GF₂ and violently boiling LF₂. 4. LF₂ containing some entrained GF₂ plus a certain amount of boiling from surfaces. This flow will continue until the tank in the vehicle is filled. <p style="text-align: right;">(U)</p>

Table V. Continued (U)

Operation	Propellant Escape or Contamination Sources
Topping off	<p>5. A short interval when flow ceases because of closure of the AGE shutoff valve. This interval lasts until the vehicle fill and drain valve closes and purge valves are opened.</p> <p>6. Reverse flow of boiling LF_2, GF_2, and purge gas (GHe) from the fill and drain line on the AGE side of the vehicle fill and drain valve to the disposal or recovery facility.</p> <p>It is highly probable that flow during periods 1 through 3 and perhaps the first part of 4 will not be at a constant rate, nor will the pressure remain constant. Pressure and flow pulsations are to be expected.</p> <p>At the end of the fill cycle, the AGE shutoff valve is closed, then the vehicle fill and drain valve, followed immediately by opening of the fill line drain and purge valves. If the last two do not open successfully with the main valves closed, a pressure rise can be expected which could lead to bursting of the line containing the trapped liquid. An uninsulated 2-in. line (heat transfer of 600 Btu/hr/ft) will probably burst (200 psi) in less than 10 sec.; a vacuum jacketed line of similar rating would not reach this point until 5 to 10 min. had passed. Rupture would cause a spray of rapidly boiling fluorine in all directions.</p> <p>During the topping of the propellant tanks, it is likely that both LF_2 and LH_2 will be transferred at the same time. Simultaneous leaks in any portion of the system will lead to the possibility of the two propellants mixing. If both systems leak into the interior of the vehicle, it is likely that ignition would occur, resulting in severe damage to some components and possible destruction of the vehicle. Slow leaks might be purged away before reaching the other propellant, especially if there is a barrier between the areas around the LH_2, and LF_2 tankage.</p> <p style="text-align: right;">(U)</p>

Table V. Continued (U)

Operation	Propellant Escape of Contamination Sources
Coupling disconnect	<p>Two situations are possible: (1) <u>disconnect following ground checkout and flow operations.</u> The coupling and fill and drain system has been completely warmed to ambient temperature and thoroughly purged using an inert gas until no liquid oxidizer is present. Only trace amounts of oxidizer present is absorbed from or in trapped areas, and then at very small concentrations. Aerosol contamination is possible during the interval between disconnect of coupling and sealing of coupling halves for storage. The degree of contamination is small because of selection of proper weather conditions, use of purges on the elements, and use of protective shrouds. (2) <u>disconnect at time of launch.</u> Residual oxidizer remaining in interconnecting lines as a result of drain and purge system failure could cause a spray of rapidly boiling fluorine from both the vehicle and the AGE oxidizer fill and drain lines. The maximum quantity is that which could be trapped between the vehicle and AGE fill and drain valves. Although localized surface reactions and the possibility of fire is ever present, the highly turbulent and reactive launch vehicle first-stage exhaust products are of a potentially far more significant nature. The rapid retraction of the oxidizer fill and drain umbilical boom will result in the rapid dispersal of residual oxidizer. This, coupled with the motion of the vehicle and the air movement caused by the exhaust products, should result in the very rapid evaporation, dilution, and dispersion of the residual oxidizer.</p>
Unloading vehicle oxidizer	<p>If vehicle launch is aborted following filling of the vehicle with propellant, it will be necessary to unload these propellants. Three possible conditions exist:</p> <ol style="list-style-type: none"> 1. Extreme vehicle hazard -- a condition (propellant leakage, uncontrollable pressure rise, fire, and so forth) exists that could, at any instant, result in a catastrophe. <p style="text-align: right;">(U)</p>

Table V. Concluded (U)

Operation	Propellant Escape or Contamination Sources
	<p>2. Possible vehicle hazard -- a malfunction or abnormal condition exists that is of unknown degree or potential effect (failure of booster engine to ignite, failure to receive a component or system signal needed to launch the vehicle, and so forth).</p> <p>3. Nonvehicle imposed aborts (poor weather, mission cancellation, range problems, and so forth).</p> <p>The conditions imposed by the first two situations suggest the rapid removal of all propellants from the vehicle to a safe storage area so that the hazard can be reduced and hopefully eliminated. The problems of propellant escape that might arise during the emptying are the same as those during fill.</p> <p>The situation of condition 3 impose no requirements for rapid or simultaneous propellant unloading. The problems are, therefore, no different from those associated with filling the vehicle.</p> <p style="text-align: right;">(U)</p>

2. EFFECTS OF FLUORINE ESCAPE AND/OR INBOARD LEAKAGE OF CONTAMINANTS

(U) Other than toxicological effects, the problems associated with the escape of fluorine from the connector and associated apparatus are of three main categories. These are as follows:

- a. Local effects on structural materials through which the escape takes place.
- b. Effects on materials upon which the escaping propellant impinges.
- c. Problems which may arise if both LF_2 and LH_2 escape at the same time.

(U) Inboard leakage of contaminants, possibly through the same pathways as that of F_2 escape, can again result in several effects as follows:

- a. Local damage on structural materials through which the contaminant passes.
- b. Degradation of other structural materials in the propellant system through corrosion caused by the contaminant.
- c. Interaction with the propellant to degrade the latter or cause unsafe conditions to develop.
- d. Clogging or otherwise restricting the proper functioning of the system and its components.

(U) In many cases, it may be expected that inboard leak of contaminants and escape of fluorine would be combined (though usually not at the same time). This would usually result in a considerable increase in magnitude of the effects. The areas included in this discussion are ones that have not been carefully studied. In most test programs, leak-tight systems are a prerequisite. If leaks develop, their effects are generally not studied; instead, the system is shut down until the leak is corrected.

(U) Nevertheless, some observations have been made as to what were the effects of the leaks, and some data collected in other studies can be applied to the leak problem. These are discussed below, and the remaining information voids are pointed out.

- (1) (U) Local Effects on Structural Materials Involved in the Leak Path -- Nonquantitative observations have been reported in which the claim is made that fluorine has the property of increasing the diameter of extremely small leak paths. It is claimed that minute openings through metal-metal and metal-Teflon seals, which will pass CH_4 but not GN_2 , will be large enough to allow passage of GN_2 and GF_2 after a period of exposure of F_2 . Like observations on integral metal structures (such as porosities in castings) have not been reported.

(U) The observations were not sufficiently controlled to determine whether leakage in only one direction was possible, or whether there could have been a breathing action with alternate attack by fluorine and airborne contaminants caused by H_2O reacting with F_2 . Nor were other conditions defined, so that individual effects of pressure difference, temperature, time, initial diameter of opening, materials, and so forth, could be isolated.

(U) If potential contaminants, especially water, are drawn inward, the behavior to be expected is related but not identical. First, if LF_2 is present or added shortly thereafter, the formation of ice crystals, which are shock-sensitive with LF_2 , is assured.

Moisture in the presence of ambient temperature, GF_2 , reacts rapidly to yield HF. The presence of HF will increase corrosion rates. In addition, if the HF then dissolves in LF_2 , it makes the now impure LF_2 an electrical conductor. This introduces the possibility of electrochemical corrosion. Electrochemical corrosion in fluorine has not been investigated. A mixture of HF and CTF is a sufficiently good electrolyte to be considered for fuel cell applications, and there are isolated but sufficient reports of electrochemical corrosion in impure LF_2 (Reference 25) to indicate that the effect can be serious.

(U) If alternate passage of materials both into and out of the system at the same opening (breathing) takes place, there generally are reactive substances available on the receiving side to combine with the substance passing. Thus, as H_2O enters, there is sufficient GF_2 nearby to react immediately and the HF formed then attacks the immediately adjacent structure. For the reverse situation, the same reactions occur when F_2 passes outward. This appears to be the best explanation of observations of certain occurrences at the Douglas Fluorine Flow Facility. Corrosion is confined to the immediate area of the leak and is fairly severe. The maximum distance from the leak that corrosion occurred was about eight inches.

(U) One type of danger that has not yet been explored is that posed by metal fluoride hydrates. Most metal fluorides are efficient absorbers of moisture to form hydrated crystals of various stoichiometries. Initial findings at Douglas indicate that these hydrated compounds are not impact sensitive in LF_2 .

(U) A system in which leak paths increase in size with time is much more dangerous than one in which leak path size remains constant. Small leaks can be neutralized by purges or vents, but if the passage size grows, such remedies will not suffice. Accurate definition of this situation is required to preclude the possibility of potentially hazardous or catastrophic results.

- (2) (U) Effects on Nearby Structural Materials -- The effects to be studied in this area will depend to a large extent on the rate at which the active materials (F_2 outward or H_2O inward) reach the materials in question. The majority of structural metals are almost unaffected by elemental fluorine, either liquid or gaseous. Static corrosion rates in pure fluorine are found to be less than 0.1 mil/year.

(U) Frost with GF_2 and LF_2 are impact sensitive, although tests have not completely defined all variables.

(U) The results of the studies conducted on frost -- F_2 impact sensitivity with an ABMA impact tester are presented in Table VI.

The specimens were as shown in Figure 36.

Table VI

100% GF ₂ on Frost*			FLOX-20 on Frost*			LF ₂ on Frost* LF ₂ at -320°F	
Specimen Number	Approx Temp (°F)	Impact Reaction	Specimen Number	Approx Temp (°F)	Impact Reaction	Specimen Number	Impact Reaction
1	-300	No	1	15+15	No	1	Yes
2	-275	No	1	15+15	No	1	Yes
3	-250	No	1	15+15	No	1	Yes
4	-225	No	1	15+15	No	1	Yes
5	-200	No	1	15+15	No	1	Yes
6	-175	No	1	15+15	No	1	Yes
7	-150	No	1	15+15	No	1	Yes
8	-125	No	1	15+15	No	1	Yes
9	-100	Yes	1	15+15	No	1	Yes
10	-75	Yes	20	15+15	No	20	Yes

*All impacts at 72 ft-lb. (U)

(U)

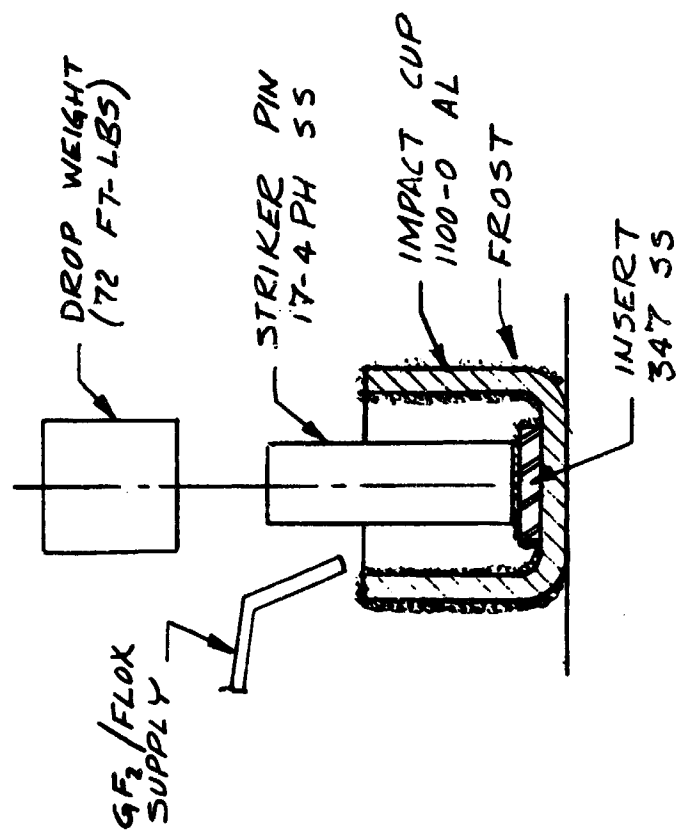


Figure 36. Frost-Fluorine Impact Sensitivity Test Setup

(U) Thus far, in the discussion of the effect of escaping F_2 on surrounding structures, we have focused solely on metals. But it is a rare structural device that does not contain other materials. This includes structures both within or outside the vehicle. Many of these other materials are organic; plastics, wood, paint, lubes and greases, potting compounds, paper, etc. Others include concrete, glass, fired clay and other similar substances. Many of these substances are basically spontaneously reactive with fluorine. But at this point, another void becomes apparent. Under what conditions will such spontaneous reactions start? There is almost no information about the fluorine concentration, the temperature, or the time factor at which various incompatible materials are susceptible to reaction. It has been determined at Douglas that Mylar and H-Film plastics are stable to 100% GF_2 at room temperature and to LF_2 at $-320^\circ F$ in static exposure. However, both are shock sensitive. Mylar reacts 100% of the time in LF_2 at a 72 ft. lbs. drop; H-Film reacted 2 out of 9 times under the same conditions. Certain rubbers (red gum, neophrene) have been used in contact with GF_2 at up to $200^\circ F$ for extended time periods, generally without any sign of violent reaction, although hardening to a non-elastic solid is generally noted. Identification of the major parameters which control initiation of reaction with fluorine is seriously needed. Only with such data can proper and safe design be applied for areas which could see accidental short exposure fluorine releases

- (3) (U) Simultaneous Escape of Propellants -- Spontaneous fluorine reactions can be expected if fluorine and hydrogen simultaneously escape from their transfer lines. It is obviously a very serious problem, but the amount of data to confirm this statement is negligible. The following observations are available:
- (a) Very pure GH_2 and very pure GF_2 have been mixed in a special apparatus without igniting spontaneously. The mixture detonated when purposely ignited.
 - (b) All engine tests involving fluorine and hydrogen ignited spontaneously. Hard starts are very rare.
 - (c) Injection of LF_2 into the GH_2 ullage of an LH_2 tank did not result in a spontaneous reaction. A violent reaction was then experienced. It was hypothesized that the LF_2 froze, dropped thru the LH_2 until it came to rest at a point where there was a heat leak (closed valve body).
 - (d) An accidental occurrence in which plumbing to cylinders of GH_2 and GF_2 sprang simultaneous leaks about 15 feet apart occurred. There was no ignition, although the odor of F_2 was quite prominent in the neighborhood of the H_2 leak.

(U) The problem is complicated by the fact that the system is not simply F_2-H_2 , but in reality $F_2-H_2-O_2-N_2$. Special mixing tests would be of interest as a method of rapidly determining the scope of the hazards. This must include basic data related to ignition limits and explosion limits of $F_2-H_2-O_2-N_2$ mixtures.

3. TOXIC RELEASE AND DISPERSION

(U) While the information required to determine the site and range operational constraints would require an evaluation of the whole system, certain useful conclusions concerning the relationship of QD leakage to toxic spill criteria may be arrived at by application of empirically derived atmospheric dispersion formulas. The purpose here is to determine the contribution of the fluorine oxidizer credible release modes of the QD to the potential toxic hazard risk to range personnel and to the population outside the boundaries.

(U) Two general types of release modes must be considered:

a. Hot Release

Major spills will probably produce a hot reaction on contact with oxidizable material. The highly reactive character of fluorine oxidizers will produce fires with oil, grease, asphalt, plastics, rubbers, adhesives, paints and vegetation. Limited reactions can occur even with sand, gravel, limestone, etc.

b. Cold Response

Large sized spills which do not ignite materials by contact with either liquid or gaseous oxidizer, are defined as cold releases.

Because of the cryogenic temperature ($-306^\circ F$) of fluorine, high flash-off rates would be expected, due to the difference in temperature between the LF_2 and the contacting surface. However, the gas produced will be more dense and tend to hug the ground for a time, flowing along the low spots in the terrain. This is a specially hazardous condition during times when wind movement is low and thermal gradients are absent. Further complications in defining rate of boiloff (emission or source strength) arise, depending on the character of the surface upon which the spill occurs. Boiloff rate will be highest from surfaces of high heat capacity and conductivity. Reduction in rate will result where puddling, containment, or contact with materials of low heat capacity or conductivity occurs.

(U) Considerable theoretical and empirical work exists which attempts to predict mathematically the air pollution hazard in terms of the mode of release, quantity released, distance downwind, and various meteorological and topographical variables known to exist in a test area. Beginning with the atmospheric turbulent diffusion equations for

continuous and point sources developed by Sutton (Reference 26), a number of extensive experimental programs were initiated to improve the predictability.

(U) Some of the more favored range dispersion equations applicable to toxic rocket propellant releases have evolved out of Projects Prairie Grass (Reference 27) Ocean Breeze and Dry Gulch (Reference 28), Sand Storm (Reference 29), and a Travelers Research Center study (Reference 30).

(U) Statistical methods were employed to simplify the prediction equations and obtain data upon which reliability figures could be stated.

(U) For predictions of fluorine oxidizer turbulent diffusion arising as a consequence of the credible release modes of a QD associated with a flight vehicle, the behavior of both quasi-instantaneous and continuous point sources must be known. A leak developed during fueling or off-loading of a propellant tank is probably best described by considering it a continuous point source if the release time extends for a period of 10 to 30 minutes and occurs at a low level near the ground. A rapidly occurring relatively large cold spill which has been contained and boils off at some average rate over a similar period of time at ground level may also possibly be treated as a continuous point source.

(U) The prediction equation favored for continuous point sources, developed out of Projects Ocean Breeze and Dry Gulch, has the following form:

$$C_p/Q = K X^a U^{-b} \sigma(\theta)^c (\Delta T + k)^d \quad (21)$$

Where:

C_p = peak concentration at a given downwind, (milligrams/cubic meter, or ppm by volume)

Q = source strength (grams/sec., lbs./min., etc.)

\bar{U} = mean-wind speed (knots, or miles/hr.)

X = distance, downwind of the sources (meters, feet or miles)

$\sigma(\theta)$ = standard deviation of the wind direction (degrees azimuth)

ΔT = difference between the temperature at two levels above ground. (A negative ΔT indicates a decrease of temperature with height. The quantity k is added to ΔT to avoid raising a negative number to a power)

$K/a/b/$ = parameters of fit (estimating equation co-efficients)
 c/d determined by least-squares regression techniques

(U) These factors operate in the following way to describe the diffusion rate.

(U) The mean wind speed, \bar{U} , is an indicator of the downwind "stretching" of the cloud emitted from a continuous source. In general, the higher the mean wind speed, the lower the concentration at a given travel distance.

(U) The standard deviation of wind direction fluctuations, $\sigma(\theta)$, as an indicator of the horizontal rate of mixing. As the value of $\sigma(\theta)$ increases, the rate of horizontal mixing increases and the concentrations decrease. Further, as the cloud grows in size, smaller eddy sizes become increasingly less effective in diffusing the cloud. In practice, this means that smoothing the wind direction data fluctuations before computing the standard deviation of the wind direction tends to increase its prediction "efficiency" for the travel distance of interest here.

(U) The vertical temperature gradient, ΔT , is an indicator of the vertical rate of mixing. If the temperature decreases with height, the rate of vertical mixing is enhanced, and the concentrations are relatively low. If the temperature increases with height, the rate of vertical mixing is inhibited, and the concentrations are relatively high.

One quantity of immediate interest to a pollution problem is the peak or maximum inhalation level concentration, C_p , at a fixed distance downwind from the source. By definition, this is the concentration found on the axis of the diffusing cloud. Concentrations decrease as one moves off the axis of the cloud.

(U) Another factor pertinent to the development of the diffusion prediction equation is the amount of material released per unit time. The downwind concentrations are obviously directly related to this factor, called the source strength, Q . As a result, one usually speaks of a "normalized peak concentration" C_p/Q ; i. e., a peak concentration divided by the source strength.

(U) The final factor to be considered is the distance, X , downwind from the source, since the peak concentration can be expressed as a power law of downwind travel distance. In actuality, it is travel time that is important to the diffusion process rather than travel distance; however, for microscale motions such as those considered here, travel time is usually discussed in terms of mean travel time to some fixed travel distance as a function of wind speed.

(U) A large amount of experimental data was introduced into a computer program to determine the parameters of fit (K , a , b , c , d) and to test the prediction efficiency of the resulting equation.

This equation in simplest form as it is now used is:

$$C_p / Q = 0.00211 \times \bar{U}^{-1.96} (\Delta T + 10)^{4.33} (\theta)^{-0.506} \quad (22)$$

Where

C_p/Q = the normalized peak concentration in sec / m^3

X = downwind travel distance in meters

$\sigma(\theta)$ = standard deviation of wind directions in degrees azimuth, with a 15 second smoothing interval

ΔT = the temperature difference in the atmosphere between 54 ft and 6 ft altitudes, in $^{\circ}\text{F}$

(U) The number 10 was added to the ΔT term in order to avoid handling the logarithm of a negative number.

(U) It will be noted that the mean wind velocity, \bar{U} , has been eliminated from the operating formula. Because an evaluation of prediction accuracy showed that wind velocity correlates with the wind shift $[\sigma(\theta)]$, and temperature lapse (ΔT). Inclusion of \bar{U} does not improve prediction accuracy.

(U) The diffusion equation is basic to the operation of the Weather Information Network Display System (WINDS) which has been developed to control toxic propellant launch operations both at ETR and WTR (Reference 31). The system is computerized and takes into account persistency of various meteorological factors, range boundary distances from the operational site, quantities of toxic propellant being handled, etc., to determine go/no-go operational conditions.

(U) At Cape Canaveral, the WIND System is set to predict C_p/Q for a downwind distance of 1.5 miles; at Vandenberg, three different distances are used depending on the location of the launch emplacement. A sample graphical solution of the equation for fluorine is included in the Appendix IV, Figure 41.

(U) It must be emphasized that application of the WINDS equation for predicting C_p/Q is strictly limited to ground level continuous point sources of about 10 to 60 minutes duration and to downwind distances of 5 to 10 miles.

(U) In the case of a fast liquid flow from an uncoupled MANSAT QD, the source would be elevated as shown in Figure 30. The WINDS system is not presently designed, therefore, to predict downwind distances and dosages for elevated releases. Oxidizers released under these conditions could be characterized by considering them as elevated point or line sources. Some work has been done to improve the original Sutton equations for these cases (Reference 32 and 33).

(U) What rational can be adopted, then, for determining the degree of hazard risk for elevated source leakage from the QD?

(U) There are various possible ways in which a sizeable spill of liquid oxidizer might occur during fueling; the line might rupture or the disconnect coupling might inadvertently release. For example, if pumping were not in progress at the time, probably only the gaseous and liquid fluorine in the line would spill. For a 2-inch diameter, 30 foot line, completely full of liquid fluorine, this would result in spilling only about 62 pounds of fluorine.

(U) For the situation where a break occurs, or the QD becomes uncoupled during a filling operation, much more fluorine would be released. Assuming a pumping rate of 20 pounds/second, and delay of 30 seconds before initiation of AGE valve closure to cut off the supply, 600 pounds of fluorine would be spilled.

(U) Although there have been several studies made of the dispersion behavior of contaminant clouds or puffs resulting from a leak or spill, it is not possible to apply them accurately to the spill situation described above, as the oxidizer spill will be occurring at a considerable distance above the ground.

(U) The shape of the cloud developing downwind is difficult to predict since it will depend on one or more of the following factors:

- (1) The flow forces set up by the oxidizer jetting may cause the transfer line to whip around in several directions.
- (2) Spreading may occur due to impingement on adjacent vehicle or AGE surfaces.
- (3) Downward streaming may occur because of the low temperature of the cryogen and its vapor density difference from that of the atmosphere.
- (4) LF_2 , or high concentrations of gas may come in contact with intervening inflammable materials and develop into a hot spill.
- (5) Meteorological conditions existing at the time including wind speed, wind direction, ambient temperature, temperature lapse rate with altitude, and atmospheric eddy currents will alter the shape.

(U) Qualitatively, the relative toxic hazard associated with instantaneous and continuous releases may be evaluated as follows (Reference 28):

(U) With the instantaneous type of release, a puff of some magnitude and original dimensions is generated which will expand and be diluted by turbulent diffusion as it travels with the wind. Turbulent eddies larger than the dimensions of the puff will cause it to meander across the terrain with but a small spreading effect as it travels downwind. With the continuous source, however, large eddies tend to spread concentrations over a wider area relative to the instantaneous puff width. Assuming no significant difference in the rate of vertical mixing and assuming an equal total amount of material is released, a lower dosage in terms

of time integrated peak concentrations will result at the center of a plume from a continuous source of 10 to 30 minutes duration, than at the point passed by the center of an instantaneous puff.

(U) Instantaneous sources released at some height or those which rise because they become hot spills produce inhalation level concentrations which differ from continuous ground sources. As a rule, the toxic hazard is greatly reduced at shorter travel distances simply because the elevated source vaults over the near areas and provides a greater depth of atmosphere between the source level and the inhalation level. This is illustrated in the Figure 37.

(U) At travel distance A, a wide margin of safety exists for the elevated source as compared to the ground-level source. At travel distance B, the maximum inhalation level concentration occurs, but this is a function of the effective source height and the atmospheric stability. In general, as with effluent from chimneys, greater source heights and greater atmospheric stabilities tend to increase the distance at which maximum concentrations occur. At travel distance C, however, little difference in breathing level concentrations will be noted between elevated and continuous ground sources.

(U) As a first approximation to evaluation the toxic hazard arising from the hypothetical elevated spill described above, the following ground level conditions will be assumed:

- (1) Instantaneous, point source
- (2) 600 pound total quantity
- (3) Ground level surface
- (4) No reaction with other materials
- (5) Density of release material equal to atmospheric density (approx.)

(U) All of these assumptions are more or less inaccurate for the release being considered, but are necessary in order to calculate a downwind dosage using existing diffusion equations.

(U) Equations similar to the following were developed in Project Sandstorm (Reference 29) to describe diffusion of an instantaneous point source:

$$\frac{E_p}{Q} = 0.180 X^{-1.59} \text{ (mean value formula)} \quad (23)$$

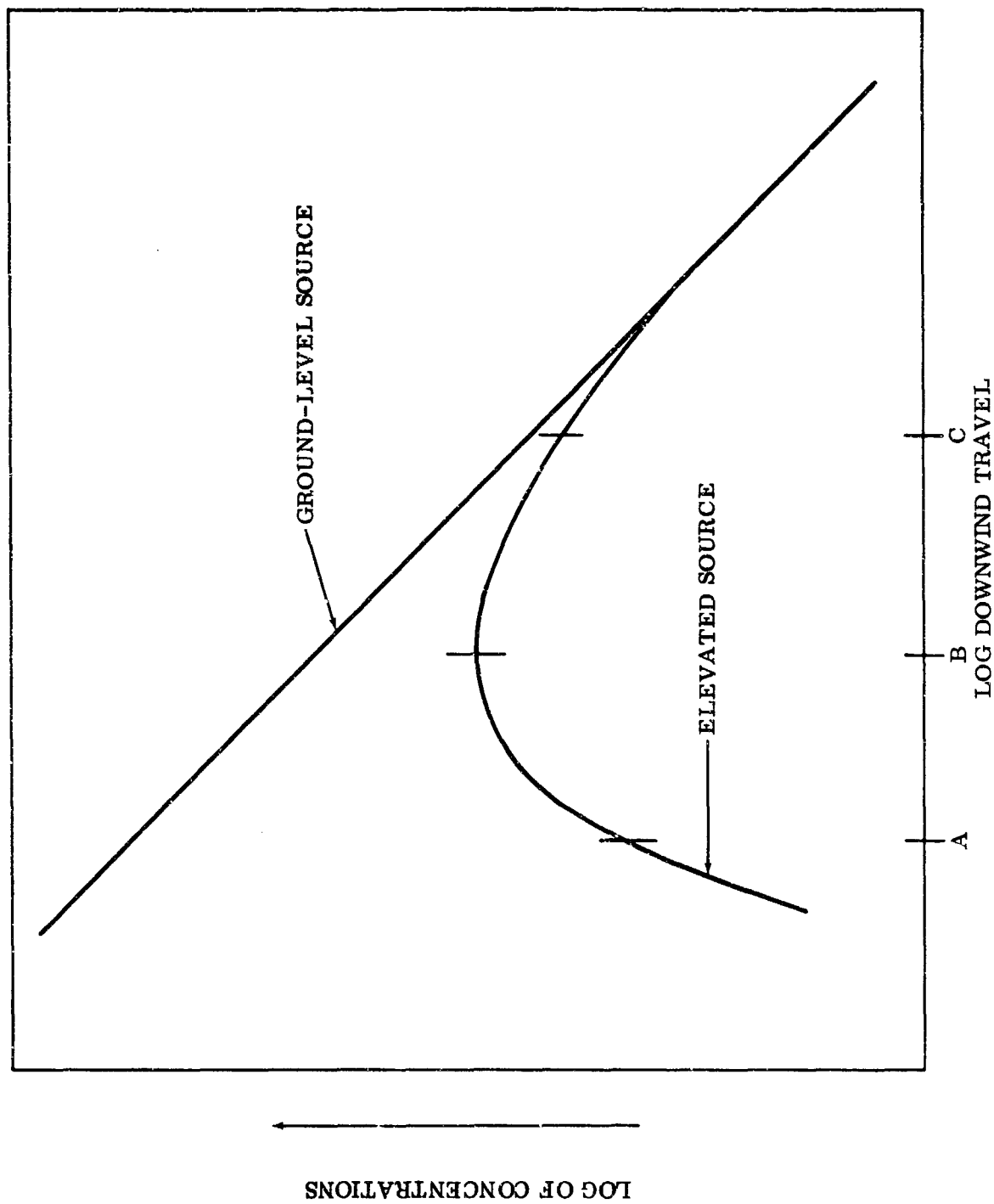


Figure 37. Ground Level Versus Elevated Source Dispersion

Where:

$\frac{E_p}{Q}$ = the peak dose normalized for source strength,
seconds/meter³

X = distance from the source in meters

0.180 = coefficient to accommodate units.

(U) At 7,000 ft downwind of a 600 lb spill, the mean dosage, E_p , will be 2.47 ppm-min. Similarly using the improved formulas (Reference 29) for the 50th and 90th percentile confidence levels the calculated dosage will be 3.4 ppm-min. and 10.2 ppm-min., respectively. In other words, the dose 7,000 ft downwind and along the centerline of the source will be greater than 3.4 ppm-min. once in every two cases, while the chance that it will exceed 10.2 ppm-min. will be one in ten. From this it is possible to gain some idea of the precision of the instantaneous point source diffusion equations available from Project Sandstorm. Actual operations will require safety factors.

(U) The above dosage calculations were made selecting 7,000 ft arbitrarily as a representative exclusion distance for safe range operations. A comparison based on emergency exposure limits (EEL's) and total integrated doses (TID's) for fluorine recommended by National Academy of Sciences, National Research Council Committee on Toxicology with the doses calculated by the Project Sandstorm method for a 600 lb, spill is indicated in Table VII.

(U) The figures indicate that peak doses at the centerline of the cloud do not exceed those recommended. The probability of exceeding the EEL with a 600 lb spill is low, particularly because the concentrations arriving at inhalation level will be less on either side of centerline.

(U) Cold spills of other magnitudes calculated with the mean value formula produce the doses listed in Table VIII.

Table VII

COMPARISON OF TID RESULTING FROM 600 LB. F₂ SPILL
WITH RECOMMENDATION TID (U)

Recommend EEL		TID (ppm-min.)	Calculated Dose (ppm-min.) Q = 600 lb
ppm	Duration (min.)		
3	10	30	3.4 (50th percentile)
2	30	60	10 (90th percentile)
1	60	60	2.47 (mean formula) (U)

Table VIII

**FLUORINE DOSES 7000 FT DOWNWIND FROM COLD SPILLS
OF VARYING STRENGTH (U)**

(Instantaneous Source, Mean Value Equation)

Q (lb/min.)	Dose (Ep) (ppm-min.)
1	0.004
2	0.008
5	0.020
10	0.041
20	0.081
50	0.204
100	0.409
200	0.818
500	2.046
1000	4.092
	(U)

(U) C_p/Q is the continuous source equation and E_p/Q in the instantaneous source equation are normalized peak doses, i. e. grams of contaminant per cubic meter (g/m^3) divided by grams of contaminant spilled or evaporated per second (g/sec). Thus, the normalized ratio is given in seconds per cubic meter (sec/m^3).

(U) To obtain parts per million per pound per minute at standard temperature and pressure, multiply C_p/Q (sec/m^3) by 1.7×10^5 and divide by the gram molecular weight of the oxidizer:

To obtain:

Multiply C_p/Q (sec/m^3) by:

$\frac{\text{ppm } F_2}{\text{lb/min.}}$

4.47×10^3

$\frac{\text{ppm } ClF_3}{\text{lb/min.}}$

1.84×10^3

<u>To obtain:</u>	<u>Multiply Cp/Q (sec/m³) by:</u>
$\frac{\text{ppm OF}_2}{\text{lb/min.}}$	3.15×10^3
$\frac{* \text{ppm HF (at 70°F)}}{\text{lb/min.}}$	3.3×10^3

(U) The *average gram molecular weight of hydrogen fluoride (HF) at ambient temperature (70°F) is estimated to be 51.3. Conversion nomographs relating C_p/Q (sec/m³) to Q , source strength (lb/min.), at various peak concentrations in parts per million for fluorine and chlorine trifluoride are shown in Figures 38 and 39, respectively.

(U) The nomographs aid in assigning a numerical value of the normalized peak concentration (C_p/Q) that can be substituted into the diffusion equation most nearly describing the particular spill or leakage mode for the purpose of determining the distance downwind beyond which the EEL established for the site (or other limit) is not exceeded.

(U) For example, returning to a consideration of the hypothetical 600 lb/min. fluorine spill: If the EEL value that must not be exceeded is 10 ppm-min., what is the exclusion distance when the C_p/Q resulting from the spill is substituted into the instantaneous point source formula. Entering the fluorine nomograph, Figure 28, when C_p/Q exceeds 3.7×10^{-6} second per cubic meter, one would expect the 10 ppm level to be exceeded at the distance for which C_p/Q is predicted when the source strength exceeds 600 lb/min.

(U) Using the distance form of the formula,

$$X \text{ (meters)} = 0.340 \frac{E_p}{Q}^{-0.629} \quad (23)$$

and substituting the value for E_p/Q , 3.7×10^{-6} sec/m³, obtained from the nomograph, the distance is computed as follows:

$$X = 0.340 (3.7 \times 10^{-6})^{-0.629}$$

$$\log X = \log 0.340 - 0.629 \log (3.7 \times 10^{-6})$$

$$= 2.993$$

$$X = \text{antilog } 2.993$$

$$= 984 \text{ meters} - 3,260 \text{ feet downwind}$$

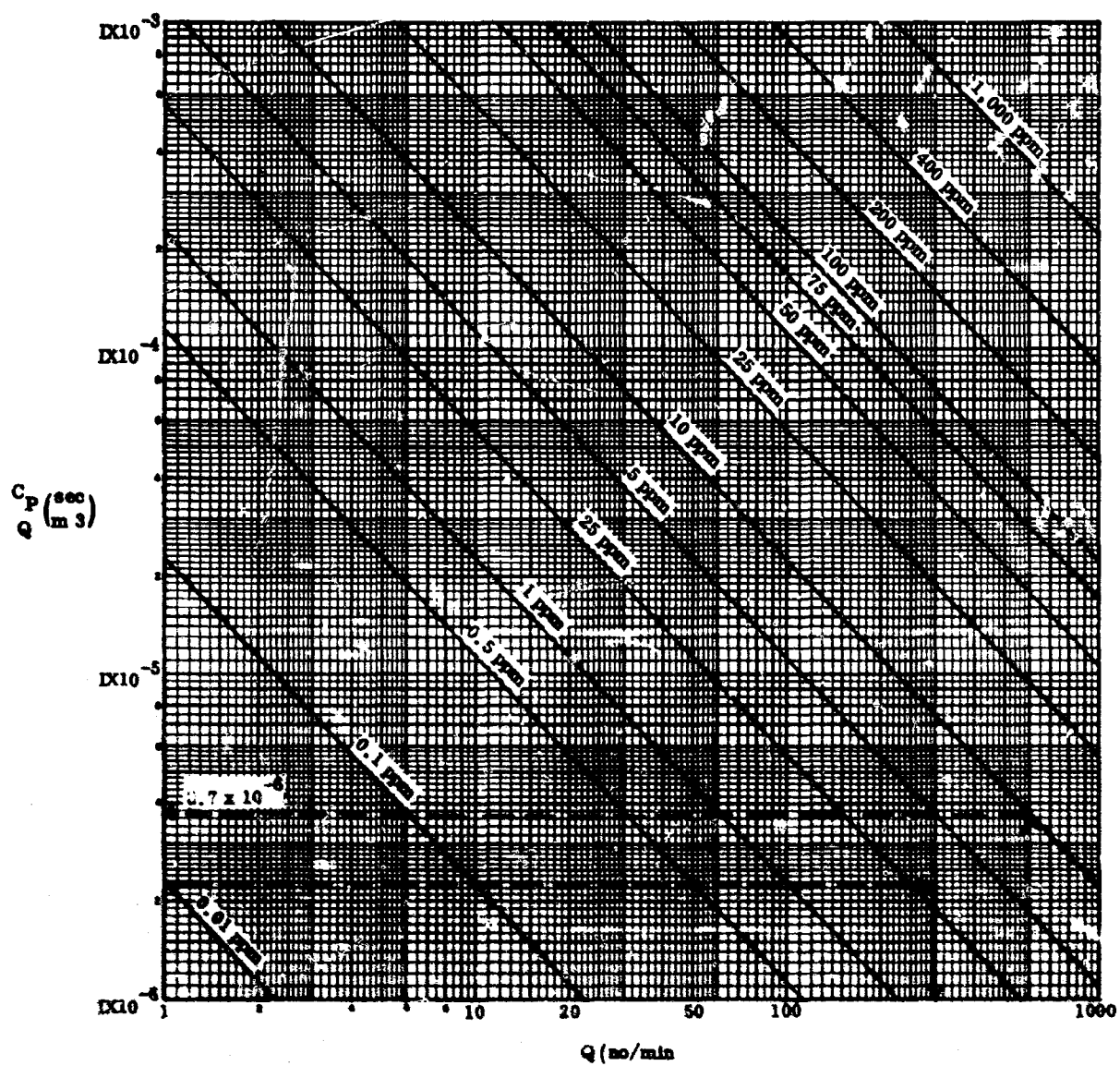


Figure 38. Conversion Nomograph F_2

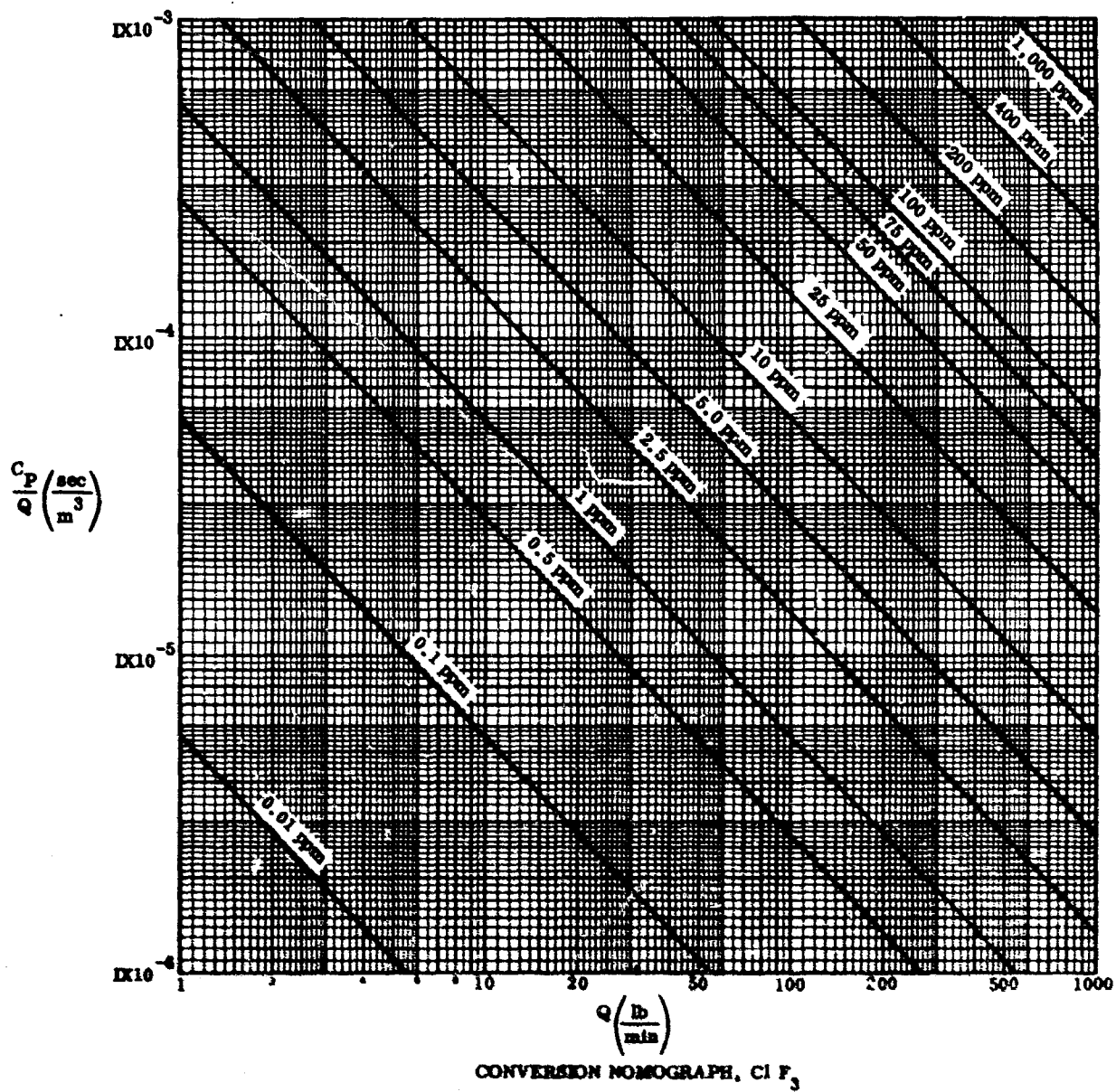


Figure 39. Conversion Nomograph CI F₃

(U) If 10 ppm-min. is the EEL for the control of the operations set by the cognizant range safety organization, and the nearest personnel are beyond 3,260 feet, the toxic hazard risk would be acceptable.

(U) For the type of cold spill where some degree of containment has been provided, and the oxidizer is evaporating at ground level, the period of time may be extended. Dispersion under these conditions will be most reliably described by the WINDS continuous point source equation currently in use at ETR and WTR.

(U) Here again, the nomograph, Figure 28, for fluorine may be used to illustrate how the toxic hazard associated with a spill can be predicted when employed along with the continuous point source equation. Suppose that with some degree of confidence, the meteorological factors registered in the WIND system yields a normalized peak concentration (C_p/Q) of 2.24×10^{-6} sec/m³ at a distance of 7000 feet downwind (the site exclusion distance) and that the fluorine peak concentration permitted is 3 ppm. What is the source strength, Q , that can not be exceeded in order that a concentration of 3 ppm will not be carried beyond 7000 ft downwind? Entering the conversion nomograph, it is found that about 300 lb of fluorine released per minute will equal a peak concentration of 3 ppm.

(U) These examples oversimplify the problem of range management. Actually the prediction accuracy in terms of a confidence limit must be determined for each situation which takes into account the meteorological forecasting accuracy and the statistical accuracy of the WINDS equation or other applicable equation. The predication accuracy of the instantaneous point source equation has been discussed previously.

(U) The accuracy of WINDS equation for continuous sources is somewhat better. Briefly, it appears that multiplying the C_p/Q obtained from the equation by 2 gives 86% confidence that the predicted peak concentration will not be exceeded. The same confidence level is achieved for the predicted distance by multiplying by 1.4.

(U) During the foregoing discussion to explore the various possible most credible release modes involving a QD and its associated AGE, it was assumed that personnel would be excluded from the site. Furthermore, the additional assumption of a 30 second delay before cut-off of the AGE valve closure resulting in a 600 lb/min. emission (at the rate of 20 lb/sec.) is probably longer than would actually be the case during a blockhouse monitored, critical transfer operation.

(U) It is reasonable to conclude therefore that the toxic hazard risk under these conditions is quite low, if the operation has been placed on a site with sufficient distances to the nearest range industrial complex, and to the nearest populated areas.

(U) There is some concern among various knowledgeable range safety personnel concerning the difficulties of prediction in all cases (Reference 34). More data on higher atmosphere behavior, including wind deviations, speeds, and particularly the deviations brought about by upper atmosphere wind shear are needed to improve prediction accuracy.

(U) At ETR, an additional 500-ft tower has been erected for the purpose of gathering such data in order to correlate it with that already feed into the WINDS system from lower altitude equipment.

(U) Finding adequate "weather winds" for launch purposes may become a problem. Diurnal variations in wind velocity, deviation, and temperature lapse in coastal areas limit the times when certain types of launches could be made. With on-shore breezes, $+1^{\circ}\text{F}$ vertical temperature lapses, and standard wind shift deviations of less than 10° , the WIND system prediction may not permit a launch operation to proceed.

Section IV

ESTABLISHED CRITERIA

(U) Summary of the design criteria established in the preceeding sections of this report are summarized here for the specific requirements imposed by the MANSAT vehicle concept presented in Section 2. 2. These criteria have provided for maximum flexibility in design and operation whenever possible, but not to the detriment of the MANSAT concept.

(U) The design objective for a coupling servicable in a system containing any of the fluorine-related oxidizers must be a leakproof separable-fluid connector that can be easily cleaned and kept free from contamination. The coupling must be adequate for transferring oxidizer from an AGE storage facility to a flight vehicle under potential temperature, vibration (induced either from the vehicle or servicing tower), wind, moisture, dust, etc. environments. It must be suitable for draining the oxidizer from the vehicle under the potential environments should a launch is abort after the oxidizer has been loaded. It must be capable of remaining attached until vehicle launch is committed and then separating cleanly and reliably.

(U) Because of the highly reactive and toxic nature of the oxidizer, the oxidizer should not be loaded into the vehicle until all tasks requiring personnel in the immediate area have been completed and the personnel evacuated. All operations involving oxidizer transfer, draining of the vehicle tank if required, draining and purging of the transfer line after transfer, and separation of the coupling at time of launch should be done by remote control.

(U) Facility and Regulatory agencies place further restrictions on the oxidizer loading and the vehicle launch activity. These regulations provide protection of employees, civilians, plant and animal life, and property on and off the site in case of inadvertent release of fluorine. The Weather Information Network Display (WIND) system controls launch operations involving toxic propellants at ETR and WTR. The system considers persistency of meteorological factors, range boundary distances from the operational site, quantities of toxic propellant being handled etc. to determine go/no-go operational conditions. As a specific application of these restrictions, the oxidizer system for the MANSAT vehicle has two separate flow paths between the AGE and the vehicle to provide fill and drain and vent.

(U) For presently conceived vehicles, there is no apparent reason to require remote connection capability. This capability would greatly increase the complexity of the design, lowering the reliability of the device and increasing maintenance problems.

(U) The following requirements, applicable for the MANSAT system as well as for any other system using a fluorine oxidizer, consider operations, flow, and mechanics.

1. GENERAL

- a. (U) The coupling should be manually connected and capable of providing positive indication of being mechanically latched.
- b. (U) The coupling must be designed for fly-away as well as for manual disconnect operations.
- c. (U) The fly-away disconnect provision must be highly reliable. (A redundant release provision may be the appropriate provision.)
- d. (U) The coupling should not provide oxidizer flow control or fluid shutoff provisions. (No integral valves would be required.)
- e. (U) The coupling must be capable of sealing against leakage in excess of 10^{-4} SCIM's when subjected to operational limit conditions (system proof pressure, vacuum, ambient, and cryogenic temperatures).
- f. (U) The coupling must adequately provide for draining and purging of wetted fluid lines between the vehicle and shutoff-isolation valves of the AGE oxidizer.
- g. (U) Provisions are required for the instantaneous determination and display of oxidizer concentration in the purge gas from the vehicle/AGE disconnect purge and drain system.
- h. (U) External leakage through the coupling seal must be safely disposed of through a duct.
- i. (U) Provisions are required for detecting external leakage through the coupling seal before the leakage could become damaging or hazardous.
- j. (U) The vehicle half of the coupling must withstand repeated use without damage or functional degradation and without needing peculiar or extensive maintenance or rework. It must be designed to guard against potential damage when not in use.
- k. (U) It is desirable that the ground (AGE) half be capable of repeated use although this half could be expendable if required.
- l. (U) The coupling must be capable of carrying all externally applied loads (wind, propellant weight, thermal stresses, etc.) without degradation to the coupling-to-coupling seal and without rendering the coupling inoperable.

- m. (U) The coupling halves must be sealed when not in use to prevent mechanical and chemical damage.
- n. (U) The coupling must be adequately isolated or otherwise protected to prevent formation of ice or frost which could potentially result in a coupling malfunction (freezing of the quick-release mechanism, etc.)

2. DETAIL DESIGN

- a. (U) Avoid the use of bellows with welded nested configuration because this configuration is difficult to clean and impossible to inspect for contamination. Make the bellows from either seamless tubing or from butt-welded wrapped sheet that is 100% X-rayed. Avoid possible hidden contamination in this weld. Bellows must be made from oxidizer-compatible materials capable of being formed into convolutions and have good fatigue properties throughout the potential temperature ranges.
- b. (U) Eliminate all possible dead-end passages.
- c. (U) Make the interior of the coupling smooth and free of cavities that could collect contaminants.
- d. (U) Make wherever possible all interior surfaces exposed to the oxidizer capable of being visually inspected.
- e. (U) Minimize pipe and tubing runs. The shortest length will have the smallest area and, correspondingly, the lowest potential source of contaminants. Minimize the number of tees, courses, elbows, and other fittings that generate and trap particles. Use manifolds wherever possible.
- f. (U) Eliminate all close-fitting dynamic parts capable of generating contaminant particles.
- g. (U) Avoid exposing threaded joints to the oxidizer. These joints form a trap for contaminants and can generate particulate contaminants during mating of the threads.
- h. (U) Avoid threaded connections. If impossible arrange them so that the flow does not scrub particles out of the threaded crevices and carry them into the vehicle.
- i. (U) Place gaskets or seals to permit minimum contact with the bulk of the working fluid.
- j. Design critical sealing surfaces to have some inherent protection (e.g. recessed surface) from damage because of handling during coupling mating.

- k. (U) Provide a pilot for aligning the mating halves of the coupling before the primary seal is engaged.
- l. (U) Avoid the necessity for rotational indexing of the coupling at the interface between the two halves.
- m. (U) Eliminate all loose hardware when the coupling separates (seals must be retained).
- n. (U) Use only clean, bagged, and sealed components to assemble the system. Inspect the bags for the presence of foreign matter.
- o. (U) Preclude moisture (water vapor, frost, and ice) from inside and adjacent to components.
- p. (U) Perform assembly and disassembly in environmentally controlled areas commensurate with the degree of cleanliness in the components being used.
- q. (U) Minimize the need for assembly and installation of fittings on a component after it has been fabricated and cleaned.

3. MATERIALS

- a. (U) Choose materials on the basis of the best possible compatibility with the oxidizer, consistent with the function of the part.
- b. (U) Require testing of the material with the oxidizer before using it in a component if there is doubt about compatibility.
- c. (U) Avoid the use of platings and coatings on metals wherever possible.
- d. (U) Avoid the use of nonmetallic materials in areas exposed to oxidizer flow. If Teflon is used as a static seal for the storable oxidizers, use it in small sections, closely surrounded by metal, and with minimum surface area exposed to the oxidizer. Teflon may also be used for a secondary seal if the cavity between the primary and secondary seals is either vacuum scavenged or purged with an inert gas.
- e. (U) Do not use lubricants or pipe compounds on joints in the fluid systems.
- f. (U) Do not use soft or stringy valve stem packings requiring periodic replacement; they are gradually deposited in the fluid stream.
- g. (U) Investigate before using two different metals in direct contact with each other whether they are compatible from the galvanic corrosion standpoint.

- h. (U) Use nonporous castings (if castings are necessary) free of sand and other foreign materials. Porous castings (particularly aluminum and bronze) should not be used because they are difficult to clean. All castings should be X-rayed or dye-penetrant inspected.

Section V

FUTURE WORK

(U) The results of this Phase I study indicate the design, fabrication, and testing of a fluorine quick disconnect (QD) can be undertaken with confidence that it will serve its intended purpose. The established criteria, design sketches, and calculations suggest a way of verifying these findings: by designing, fabricating, and testing the coupling sketched in Figures 4 and 5. The objective of the testing program will be to verify that these study findings are valid by demonstrating the quick disconnect in a simulated fluorine transfer system.

(U) Although several information voids were identified in this study, none appears to affect the design, fabrication, or testing of this quick disconnect. In summary, these voids are as follows:

1. (U) Fluorine oxidizer leak detection and measurement devices for use with an operational QD are nonexistent.
2. (U) Data on the corrosion of metals by galvanic couples in LF_2 are lacking.
3. (U) Quantitative data on the effects of fluorine oxidizers passing through small passages or porous materials are lacking. Qualitative data indicates that small leaks, which are likely to develop during a fluorine QD development program, should not present any problems.
4. (U) Data on the interaction of F_2 , H_2 , air, and H_2O vapor are needed to more clearly define system and component leakage limitations. The development of a fluorine quick disconnect (QD) will occur in the absence of H_2 and under conditions where H_2O vapor can be very carefully controlled.

1. DESIGN OF A TEST QD

(U) Because of the potential hazards of fluorine leakage, the interface seal between the halves of the coupling is perhaps the most critical area in the design. The development program will include determination by testing of the effect on leakage of varying the seal materials and seal preloads, and of using single, as opposed to double, sealing surfaces. The design phase comprises the steps in the following paragraphs.

a. Preliminary QD Specification Drawing

(U) This step identifies in Military Specification format the specific requirements that must be met in the design and testing of the QD.

b. Conduct a QD Sizing Analysis

(U) A sizing analysis will be conducted in conjunction with Step 1 to determine the permissible pressure drop and the required flow area through the coupling. A purge flow analysis of the propellant transfer line will be conducted to determine the required sizing and location for vent and purge ports.

c. Sketch Concepts

(U) Concurrently with Steps 1 and 2, sketches will be made of the QD concept in Figure 4 and of those related system components required to conduct demonstration tests. The QD concept will be refined. After the sketching and evaluating, the most likely fill-and-drain system concept for MANSAT will be selected and further efforts will be confined to developing it into a final design.

d. Conduct Design Analyses

(U) Design analyses will verify the structural integrity of the QD and its related systems and will provide assurance that design requirements will be met. The following analyses are required:

- (1) Strength analysis.
- (2) Thermal effects analysis.
- (3) Seal Preload analysis.
- (4) Pressure effects analysis.
- (5) Fill-line external loads analysis.
- (6) Unlatching loads analysis.
- (7) Anti-icing shroud purge flow analysis.

e. Finalize Layouts

(U) Concurrently with Step 4, the following layouts for the selected concept will be completed:

- (1) QD coupling.
- (2) Anti-icing shroud.

- (3) Simulated transfer system for test.
- (4) Purge and drain systems for test.
- (5) Latch actuation system for test.
- f. Prepare Test Plan
 - (U) Concurrently with Steps 4 and 5, a test plan will be prepared based on the requirements in Subsection 2, Test Program Requirements, of this document.
- g. (U) The following tasks will be included:
 - (1) Finalize the test requirements.
 - (2) Finalize the testing approach.
 - (3) Identify the test equipment required.
 - (4) Prepare a detailed test plan.
- h. Submit for AF-RPL Approval
 - (U) When the above steps are completed, the layouts and test plan will be submitted to the customer for approval before preparation of detailed fabrication drawings
- i. Prepare Test Equipment Drawings
 - (U) During the interim between submittal of the layouts and test plan and the approval by the customer, drawings of equipment and fixtures for use in testing the QD will be prepared.
- j. Prepare Detail Drawings
 - (U) After approval of the layouts by the customer, detail drawings will be prepared of the QD, the anti-icing shroud, the propellant transfer line, and the vent and purge system.
- k. Prepare Test Procedure
 - (U) Concurrently with Step 9, test procedures will be prepared, providing detailed instructions for implementing the test program.
- l. Fabricate and Test QD
 - (U) A test model QD will be fabricated from detail drawings. The necessary test equipment and fixtures will be procured and fabricated. Testing will be conducted in accordance with the test procedures of Step 10.

m. Evaluate Results

(U) Test results will be evaluated, and the following items will be investigated:

- (1) Effects of varying seal parameters on leakage.
- (2) Performance of purge system in removing trapped fluid from transfer line.
- (3) Performance of shroud purge system in preventing formation of frost and ice around the QD.
- (4) Performance of latch release mechanism.
- (5) Performance of any F₂ leak detection devices that may have been used during the testing.
- (6) Effects on the QD of dynamic liquid and gaseous fluorine exposure.
- (7) Durability of the QD to undergo typical repeated connect-disconnect cycles.

(U) If, as a result of the testing program, it becomes evident that any of the criteria established in Phase I is incorrect or not applicable, the criteria will be upgraded in the final report. Also, problems not anticipated may be encountered and result in new information voids in fluorine technology. In this event, an attempt will be made to fill the voids if feasible (without embarking on a major new program.). If a void develops in a major problem, it will be so noted and the solution will be recommended for a future program.

n. Modify Design as Required

(U) In case any deficiencies in the design of the test model QD are discovered during the testing program, the design will be modified to correct the deficiencies. The detail drawings will be changed to reflect the redesign before release for fabrication of a prototype model of the QD. The specification drawing will also be updated with any changes required in the design.

o. Fabricate and Acceptance Test Prototype QD

(U) A prototype QD incorporating all design improvements discussed in the preceding steps will be fabricated. It will undergo acceptance testing, including proof pressure, leak check at liquid nitrogen temperature, and functional testing of latching mechanism. After satisfactory completion of these tests, the coupling will be delivered to the customer.

p. Prepare Final Report

(U) A final report summarizing the results of the entire program will be prepared (including all conclusions and recommendations). It will be supported by all available data pertinent to the subject of the contract. It will include hardware details and unsuccessful test results.

2. TEST PROGRAM REQUIREMENTS

(U) The primary objective of the test program is to prove that the design is functionally good and compatible with the use of liquid fluorine as an oxidizer. The program will prove system concepts, rather than qualifying detailed hardware for use on a particular vehicle. In addition, limited design data on scaling and environment will be evolved. Major areas of concern are as follows:

a. Fluorine Detection

(U) Requires further development testing to adapt existing devices to function as a fluorine indicator for the purge system and for the leak detection system.

b. Drain and Purge System Effectiveness

(U) Requires investigation to prove the concept that liquid and gaseous oxidizer can be removed from the fill and drain line to safe concentration level before the coupling is separated.

c. Separation Mechanism Effectiveness

(U) Requires demonstration testing to verify a design concept for primary separation and redundant separation methods. Although the separation mechanism chosen will not involve a new, untried concept, evaluation of such a mechanism under the particular environmental conditions of the MANSAT vehicle is required (field assembly environment, corrosive cryogenic media, etc.). Testing will serve to prove the effectiveness of the separation method with the particular seal design chosen.

d. Interface Seal

(U) Requires investigation to prove the sealing capability under the expected field conditions and the reliability when used to tank and detank with liquid fluorine after being connected and leak-checked. In addition to this demonstration testing, investigation should be made to determine the effect of varying key sealing parameters to indicate the true factors of safety involved and the capability for scaling the critical elements of the design.

e. **F₂ Compatibility**

(U) Requires exposure to typical service conditions involving F₂ to prove compatibility.

f. **Anti-icing Shroud Purge**

(U) Requires investigation to establish effectiveness of preventing the formation of ice and frost on the QD latch and separation mechanism.

3. **PROPOSED TEST PLAN OUTLINE FOR QD COUPLING TESTING**

a. **Seal and Separation Mechanism Checkout (GHe or GN₂)**

(U) After fabrication, set up test hardware in Propulsion Laboratory for a dry checkout. Modifications made as required.

- (1) Mount vehicle half in support fixture and attach blind flanges.
- (2) Install secondary (Teflon) gasket and attach ground half.
- (3) Torque bolts equally to 20% while measuring gasket deformation with a dial indicator.
- (4) Check for overall leakage with bubble bath.
- (5) Release and recouple to checkout attach mechanism and secondary gasket.
- (6) Install aluminum gasket and Teflon gasket and torque to 50%, 75%, 100%, 125%, and 150%, measuring leakage each time.
- (7) Release to checkout mechanism under load.
- (8) Repeat with a new aluminum gasket to confirm.
- (9) Repeat with the gaskets specially modified to show the effects of a single wedge, a corroded gasket, and an improperly torqued gasket.

b. **Cryogenic Functional Tests**

(U) Set up coupling with a liquid nitrogen source and a shutoff valve downstream so that LN₂ can flow through the coupling under 30 to 50 psig.

- (1) Install new gaskets and frost shield, torque to rated value, and leak check with mass spectrometer.

- (2) Flow LN_2 , record exterior temperature at 4 points, purge frost shield with helium, and leak check secondary gasket.
- (3) Shutoff LN_2 , pressurize that chamber with helium, and leak check primary seal as the coupling warms.
- (4) Flow LN_2 again to chill down coupling and separate to check mechanism while cold.
- (5) Repeat leak checks with new gaskets.
- (6) Repeat leak checks with modified gaskets to show effects of single wedge, and an improperly torqued gasket.

c. Liquid Fluorine Compatibility Tests

(U) Clean and set up coupling in the fluorine flow loop at remote test site with shutoff valves to simulate vehicle conditions and all leak detection, purge, and vent systems installed.

- (1) Install new gaskets, torque to rated value, and leak check.
- (2) Purge and passivate coupling, leak detection, and vent systems. Purge and checkout leak detection system.
- (3) Purge flow loop and recheck for leakage with helium.
- (4) Flow 250 gal LF_2 through coupling in both directions. Purge and vent trapped volume between the shutoff valves to checkout the vent system function.
- (5) Hold system under blanket purge for 8 to 24 hours.
- (6) Disassemble, check visually, and install special gaskets to show the effects of a damaged and improperly torqued gasket. Conduct these tests using gaseous fluorine to check out leak detection and purge vent system.
- (7) Replace gaskets and conduct a series of tests to evaluate liquid fluorine extraction from the trapped volume between the two shutoff valves. Use thermocouples in the lines to detect presence of LF_2 and use F_2 detector to record the concentration of F_2 in purge gas.
- (8) Flow 250 gal LF_2 in each direction and disconnect as soon as F_2 concentration is safe.
- (9) Replace gaskets and flow 250 gal LF_2 . Purge F_2 from system and disconnect QD.
- (10) Repeat latter test as time permits.

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Appendix I MANSAT SYSTEM STUDIES

(U) The following discussion of the MANSAT Vehicle System was taken directly from Reference 35.

1. FEED-LINE SYSTEM

(C) "Propellants are fed to the engine system through low-pressure ducting as shown schematically in Figure 2. Both feed lines are vacuum jacketed and contain several layers of high-performance insulation within the vacuum jacketing to reduce feed-line heating during coast. Vacuum jacketing is used to prevent the formation of liquid air around the feed lines and to reduce the heat flux into the lines during propellant-fill and ground-hold operations. Further analysis may indicate that vacuum jacketing is not required for the LF_2 feed line."

(C) "The LF_2 feed line is approximately 14 ft long. Because of its length, this line must be wet prior to engine start to avoid excessive LF_2 lag during the start transient. A wet line is maintained by incorporating a tank-mounted valve and a bleed line into the feed line. This system design is based on the results of a preliminary thermal analysis. After engine shutdown, the tank-mounted pre valve is closed, trapping liquid in the feed line. As the LF_2 , in the vicinity of the engine, is vaporized due to engine heat feed-back, a portion of the LF_2 is displaced through the bleed line into the LF_2 tank. Once the vapor bubble grows to a height which uncovers the bleed line, further heating results in surface evaporation of the LF_2 . Regression of the LF_2 in the feed line is sufficiently slow so that the line will be at least 85% full prior to engine restart. The vaporized fluorine is continually bled into the tank to prevent excessive pressure build-up in the feed line during coast. The tank valve prevents back-flow of liquid into the tank during the coast period. A shutoff valve was used for this purpose rather than a check valve to reduce the line pressure drop during engine operations. A low ΔP check valve is located in the bleed line to prevent ullage gas from entering the feed line during engine operation."

2. FILL AND DRAIN SYSTEM

(C) "The propellant tank fill system is shown schematically in Figure 2. Both tanks are filled through separate lines passing through the vehicle sidewall and into each tank. Incorporated in each fill line is a shut-off valve which seals the tank prior to launch. This valve is located close to the tank wall in the highly insulated portion of the system to keep liquid out of the fill line during coast, thereby reducing

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heat leak into the system and minimizing the trapping of unusable oxidizer. During main fill and topping operations the umbilical propellant transfer lines are connected to the vehicle fill system with quick-disconnect couplings. These couplings do not have internal poppets, thereby preventing trapping of propellants between the main shutoff valve and quick disconnect. During the filling and topping operation, the tank vent valves are open, allowing the vented gases to escape into the ground-venting system. Both engine prevalues are closed and the tank-mounted LF₂ prevalue is open during the entire filling operation. Just prior to liftoff, all valves are closed, and the fill umbilical lines are drained and purged."

(C) "According to the criteria of Reference 36, the L/D ratio (65) of the LF₂ feed lines is high enough to result in geysering during fill. This can be prevented by using the bleed line (which is already required to maintain a wet line during coast) to allow recirculation of the feed line chilldown vapor into the main-tank ullage. By proper sizing of bleed line, backflow or geysering of the LF₂ in the feed line can be prevented. The pressure buildup, due to the rapid vaporization of the LF₂ while chilling the feed line, is relieved directly into the ullage. Cursorv analysis has indicated that a 3/4 inch diameter bleed line should prevent geysering in the LF₂ feed line during fill."

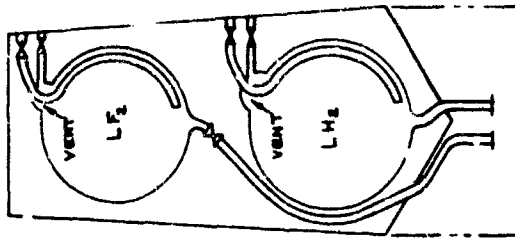
(C) "Prior to selection of the fill system, shown in Figure 2, several other concepts were considered. These are shown schematically in Figure 40. The fluorine-fill concept, shown in Configuration 1, was not satisfactory because it provides an additional heat path from the vehicle sidewall to the feed line. This heat path could create a vapor bubble at some point midway in the wet portion of the feed line. During engine start, the vapor bubble could be ingested by the engine pump. This bubble ingestion is not desirable since it may cause pump damage due to slugging and/or pump overspeed during the bootstrap period to full power. Because of the critical nature of feed line heating, which may result in improper functioning of the propellant orientation system, the philosophy for the fill-system design has been to minimize heat addition into the feed line wherever possible. The LH₂ fill-line concept of Configuration 1 was discarded because it permits an increase in heat flux into the critical sump area."

(C) "The fill-line concept shown in Configuration 2 is designed to eliminate feed-line heat shorts into critical areas as described above. However, LF₂ trapped in the fill line must be vented. The nuisance of having to vent trapped propellants, even though heat shorts are removed from the critical regions, negates any real advantage of this system over the selected one."

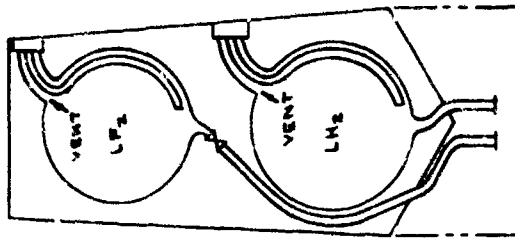
(C) "Short, closed-coupled, fill lines, shown in Configuration 3, were also considered. While minimizing trapped propellants and feed-line heat shorts, other considerations discouraged the use of this system. For example, the umbilical transfer lines must pass through the inter stage area to mate with the fill-line couplings. This is not desirable

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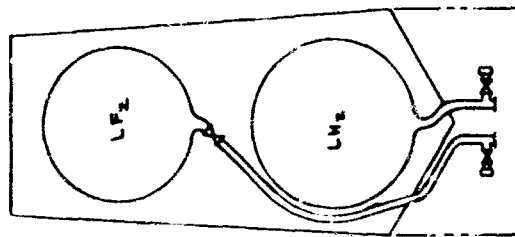
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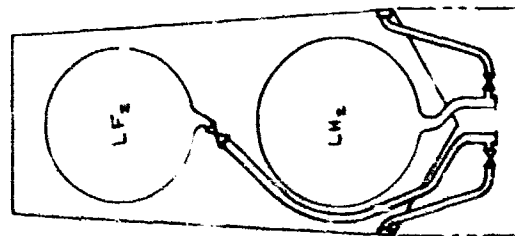
CONFIGURATION 5



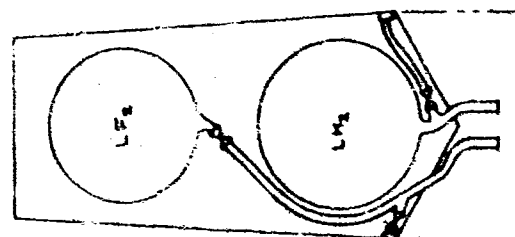
CONFIGURATION 4



CONFIGURATION 3



CONFIGURATION 2



CONFIGURATION 1

Figure 40. Candidate Fill and Drain Systems

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from interstage structural considerations. Also, the umbilical/stage propellant transfer couplings would not be visible during the filling operation. Any leakage could go undetected until a dangerous accumulation of propellant vapor existed within the confined interstage area. In addition, any propellant spillage during transfer line disconnect would also accumulate in the interstage area creating an explosion hazard."

(C) "Two schemes were evaluated for filling the propellant tank through the tank vent-line penetration. These are shown on Configurations 4 and 5 in Figure 40. These methods reduce the number of tank penetrations by combining the fill and vent lines. In Configuration 4, a concentric line is routed into one valve body which has internal porting to separate the vent and fill functions. The tanks are filled through the inner annulus and vented simultaneously through the outer annulus. This would require the development of a cryogenic concentric line and a rather complex valve. Separate vent and fill valves can be used if a manifold is used to separate the fill and vent lines prior to the valving, as shown in Configuration 5."

(C) "The total system weight for Configuration 4 or 5 should be about the same as for the selected fill method. Because there is no urgent need to reduce the number of tank penetrations, and, since the concentric fill vent system is structurally complicated, this system is not recommended."

3. VENT SYSTEM

(C) "Both tanks are vented during propellant filling through separate vent lines which are connected to the vehicle sidewall. Incorporated in each vent line is a shutoff valve which seals the tank prior to launch. This valve is located close to the tank rather than the sidewall to keep liquid out of the vent line during coast, thereby reducing heat leak into the propellant. During main fill and topping operations, the umbilical vent lines are connected to the vehicle vent system with quick disconnect couplings. The quick disconnect couplings do not have internal shutoff provisions, thus allowing emergency venting during the mission."

(C) "Since in-flight venting is not expected, the vehicle vent system will be sized to prevent tank overpressurization during the filling operation. However, the fill-time and ground-propellant transfer and venting system characteristics were not specified, and sizing analysis was not performed. Therefore, 2-inch diameter vent lines were arbitrarily selected."

4. THERMODYNAMIC ANALYSIS

(C) "This analysis includes propellant heating during propellant loading and ground hold conditions. Propellant boiloff occurs by absorbing heat from two sources (1) heat stored in the warm walls of the oxidizer

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system prior to loading with oxidizer; and (2) heat transferred through the tank walls and heat shorts from the surrounding environment. "

(C) "Propellant required to remove the residual heat in the tank walls is significant, but is only a small percentage of the weight of propellant required to fill the tanks. To chill the tank walls from an initial temperature of 100°F to the temperatures of the oxidizer, -306°F for LF₂, 62.5 lb of LF₂ are required. This represents approximately 0.40% of the LF₂ required to fill the tanks. "

(C) "The insulation technique chosen for the vehicle piping involves vacuum jacketing the propellant feed lines while leaving the fill and vent lines bare. The total heat transfer rate to the oxidizer from all of the subsystems, that is, the tank walls, line supports, fill, vent and feed lines are determined to be 12,415 BTU/hr. for LF₂, or 169 lb/hr. of LF₂. Approximately 99% of this ground hold heat leak is attributed to the tank itself, with approximately 0.3% from the combined effects of the fill and vent lines together. Heat leak through the fill and vent systems is negligible. "

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Appendix II

OXIDIZER CHARACTERISTICS

(U) A summary of the information on the characteristics, principal byproducts, and contaminants of the four oxidizers under consideration is shown in Tables IX and X.

(U) For liquid fluorine (LF_2) it may be noted that in addition to F_2 the primary reactive constituents are hydrogen fluoride (HF) and oxygen difluoride (OF_2); both products are formed from moisture contamination. Oxygen (O_2) is considered mildly corrosive and the remaining constituents are either inert or have negligible corrosive effects.

(U) For chlorine trifluoride (CTF), the most reactive constituent is also HF . Perchloryl fluoride (ClO_3F), chlorine monofluoride (ClF), and chlorine (Cl_2) are also corrosive but to a lesser degree than HF . The remaining constituents are either inert or have negligible corrosive effects.

(U) FLOX, a mixture of liquid fluorine and liquid oxygen, may contain all of the contaminants noted for liquid fluorine plus those which are common to liquid oxygen. Oxygen is produced by liquefaction of air, and its principal contaminants are nitrogen and argon, which are inert. In general, LO_2 contains a higher percentage of hydrocarbons than gaseous oxygen. When FLOX is prepared in large quantities, it is usually prepared by combining LO_2 and LF_2 . Reaction between LF_2 and the hydrocarbons may cause an appreciable increase in the HF present in the FLOX. Smaller amounts of the contaminants per unit volume should be present in FLOX than in LF_2 , and as would be expected, FLOX has been shown to be less corrosive. Rocketdyne has reported that in tests which they have conducted, the compatibility of metal materials with FLOX with concentrations below 40 or 45% fluorine is essentially the same as for liquid oxygen. With high concentrations of F_2 , the FLOX reacted with metals to a degree approaching the reactions with LF_2 .

(U) The chemical composition of Compound A is classified, and a breakdown of its byproducts and contaminants are not included in this report. However, after laboratory testing of both CTF and Compound A, Rocketdyne has reported that, in general, materials which are compatible with CTF are also compatible with Compound A, and that CTF criteria are applicable.

Table IX
LIQUID FLUORINE AND TYPICAL CONTAMINANTS - (U)

Formula	Name	Melting Point °C	Boiling Point °C	Relative Reactivity with Materials of Construction	Typical Quantities Present	Remarks
F ₂	Fluorine	-219.6	-188	Highly corrosive	98.5+%	Both liquid and gaseous states are reactive in varying degrees with essentially all materials of construction.
HF	Hydrogen fluoride	-83.1	-19.54	Highly corrosive in liquid and gaseous states	0.15%	Apparently non-corrosive in solid form. Presence of HF is generally attributable to the reaction of gaseous F ₂ with moisture from the atmosphere.
O ₂	Oxygen	-218.4	-182.96	Mildly corrosive	0.15%	Compared to F ₂ , O ₂ is only mildly corrosive to most materials of construction. A product of moisture.
N ₂	Nitrogen	-209.86	-195.8	Noncorrosive	0.45%	Inert gas
CF ₄	Carbon tetrafluoride	-184	-128	Noncorrosive	0.01%	Characterized by extreme chemical inertness at ordinary and low temperatures. A product from carbon electrodes.
CO ₂	Carbon dioxide	-56.6 @ 5 ATM.	-78.5 Sublimes	Noncorrosive	0.001%	Negligible corrosion rates with the common materials of construction. A product from impure electrolyte. Has impact sensitive reaction with LF ₂ .
OF ₂	Oxygen difluoride	-223.8	-144.8	Highly corrosive	Not Determined	Short term tests of LF ₂ containing 10% OF ₂ suggest that OF ₂ accelerates corrosion. At levels it is normally present it appears unlikely that it would have a measurable effect. A product from moisture in the F ₂ generator.
COF ₂	Carbonyl difluoride	-114	-83.1	Not identified as corrosive		Essentially insoluble in LF ₂ and apparently does not contribute to corrosion. A product from impure electrolyte.
SO ₂ F ₂	Sulfuryl fluoride	-136.7	-55.4	Not identified as corrosive		Essentially insoluble in LF ₂ and apparently does not contribute to corrosion. A product from impure HF.
SF ₂	Sulfuryl hexafluoride	-50.5	+63.8	Noncorrosive		Essentially insoluble in LF ₂ and comparable to N ₂ in chemical inertness. A product from impure HF.
SiF ₄	Silicon tetrafluoride	-90.2	-86	Not identified as corrosive		Essentially insoluble in LF ₂ and apparently does not contribute to corrosion. A product from glass (not normally present). May form from silicon in metal alloys.
He	Helium	-272.2	-268.9	Noncorrosive		Inert gas
H ₂	Hydrogen	-259.14	-252.8	Noncorrosive		Negligible corrosion rates with common materials of construction.
Metal F	Metal fluoride	-	-	Noncorrosive	Not Determined	Forms on all metals in contact with F ₂ . May be present as solid particles in the fluid as a result of being separated from the base metal. Increases electrical conductivity of the fluid and causes increased galvanic corrosion.

Table X
CHLORINE TRIFLUORIDE AND TYPICAL CONTAMINANTS - (U)

Formula	Name	Melting Point °C	Boiling Point °C	Relative Reactivity with Materials of Construction	Typical Quantities Present	Remarks
ClF ₃	Chlorine trifluoride	-83	+11.3	Highly corrosive	99% Spec. Min.	One of the most reactive of the halogen fluorides. Made from chlorine and fluorine at 200°C.
HF	Hydrogen fluoride	-83.1	+19.54	Highly corrosive in liquid and gaseous states	0.5%	Presence of HF is generally attributable to reaction of fluorine with moisture from the atmosphere.
Cl ₂	Chlorine	-103±5	-34.6	Corrosive	Not Determined	Dry chlorine is moderately corrosive to metals. Chlorine combined with water is highly corrosive to metals.
ClF	Chlorine monofluoride	-154±5	-100.8	Corrosive	0.25%	ClF is less reactive than ClF ₃ , therefore should not accelerate the corrosion rate.
ClO ₂	Chlorine dioxide	-59.5	+9.9	Not identified as corrosive	Not Determined	ClO ₂ is a very unstable and explosive compound. It is debatable whether significant quantities would be present.
ClO ₂ F	Chloryl fluoride	-115	-6	Not identified as corrosive	0.25%	ClO ₂ F is unstable and shock sensitive. Dense white fumes of HF are formed when exposed to moist air. Chemical reactions have not been studied extensively.
ClO ₃ F	Perchloryl fluoride	-146	-46.8	Corrosive	Not Determined	ClO ₃ F is a stable compound. Not corrosive to most metals in anhydrous state; but moist gas is severely corrosive to aluminum and steel.
CF ₄	Carbon tetrafluoride	-184	-128	Noncorrosive		Characterized by extreme chemical inertness at ordinary and low temperatures. An impurity from the manufacture of F ₂ .
CO ₂	Carbon dioxide	-56.6 @ 5.2 ATM.	-78.5 Sublimes	Noncorrosive		Negligible corrosion rates with common materials of construction. An impurity from the manufacture of F ₂ .
Metal F	Metal fluoride	-	-	Noncorrosive	Not Determined	Forms on all metals in contact with F ₂ . May be present in solid particles in the fluid as a result of being separated from the base metal. Increases electrical conductivity and causes increased galvanic corrosion.
						NOTE: Some explosions in ClF ₃ systems which have happened as ClF ₃ evaporates have been tentatively blamed on chlorine oxides and oxyfluorides present in the ClF ₃ .

Appendix III
MATERIALS COMPATIBILITY

1. COMPATIBILITY OF MATERIALS OF CONSTRUCTION WITH LIQUID FLUORINE

(U) Several test programs to determine the compatability of materials with LF_2 have been conducted by various research organizations. Table XI indicates the conclusions reached by researchers after a literature search and after survey trips to several organizations which had actual experience with LF_2 testing.

(U) Tables XII through XV present the results obtained from some typical test programs and indicate the relative corrosion resistance of some of the common materials. No attempt has been made to rationalize the differences in the magnitude of corrosion rates reported by the different researchers. The different values may be the result of different exposure time, different test conditions, or different methods for assessing corrosion rates. However, the relative corrosion rates of the various materials should be sufficiently valid to serve as a basis for selection of a material for a specific application.

(U) Table XVI presents the classification code representing the compatibility of liquid and gaseous F_2 with the materials listed in Table XVII, which compiles the data gathered and published by the Defense Metals Information Center.

Table XI
STRUCTURAL MATERIALS COMPATIBLE WITH LF_2 IN THE
ORDER OF DECREASING CORROSION RESISTANCE. (U)

- | |
|---------------------------------|
| 1. Monel |
| 2. Nickel |
| 3. Low silicon stainless steels |
| 4. Copper |
| 5. Aluminum |
| 6. Magnesium |

Reference 37

(U)

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(U) The following pertinent information on the compatibility of materials with fluorine and comments on fluoride films were extracted from Reference 37.

(C) Laboratory test and handling experience in recent years reveals that several metals have been used with both gaseous and liquid fluorine at room and cryogenic temperatures. Of these, Monel has the highest corrosion resistance, followed in order by nickel, low silicon stainless steels, copper, aluminum, and magnesium. The utility of these metals is based on the formation of comparatively tenacious fluoride films which tend to protect the metal from further attack. The density and adherence of the protective film is a measure of the relative value of the metal for service in fluorine; nickel and Monel will form a dense, tough coating whereas steels form loose, porous coatings and are less corrosion-resistant. High-velocity liquid flow studies are used to indicate the films that are stable.

Table XII
CORROSION OF METAL IMMERSSED IN LF_2 FOR 1 YEAR* (U)

Metal	Corrosion Rate (mils/year)
15-7 Monel	0.0010
304 Stainless steel	0.0026
Copper	0.0028
Nickel	0.0034
Monel	0.0035
410 Stainless steel	0.0630
AL 6061	0.1843
AL 1100	0.2093
TiC-120	0.2267
TiA-110	0.2806
Mg AZ-31	0.3437
Mg HM-31	0.6741
*Based on five specimens for each material. (U) Reference 38.	

Table XIII

CORROSION OF UNSTRESSED METALS IN LF_2 ,
7- TO 14-DAY EXPOSURE (U)

Material	Alloy	Corrosion Rate (mils/year)	No. of Samples
1. Magnesium	HK (coated with DOW-17)*	0.1	4
2. Stainless steel	304	0.2	8
3. High-strength steel	PH15-7 Mo	0.2	6
4. Nickel		0.2	6
5. Cupro-nickel	10% Ni	0.2	4
6. Brass	Cartridge	0.2	12
7. Stainless steel	316	0.3	8
8. Stainless steel	420	0.3	8
9. High-strength steel	AM-350-CX	0.3	8
10. Stainless steel	347	0.4	8
11. High-strength steel	AM-350-C	0.4	7
12. Copper		0.4	10
13. High-strength steel	AM-350-D	0.5	8
14. Monel		0.5	4
15. Brass	Yellow	0.5	2
16. Aluminum	1100	0.6	14
17. Aluminum	6061	0.6	15
18. Cupro-nickel	30% Ni	0.6	5
*DOW-17 -- A proprietary anodic anticorrosion conversion coating for magnesium and magnesium alloys.			(U)

Table XIII -- Concluded (U)

Material	Alloy	Corrosion Rate (mils/year)	No. of Samples
19. Magnesium	AZ-31 (coated with DOW-17)	0.6	4
20. Aluminum	7079	0.7	15
21. High-strength steel	AM-350-DX	0.7	8
22. Magnesium	HK-31	0.8	10
23. Everdur	1010	0.8	6
24. Magnesium	AZ-31	0.9	6
25. Titanium	A110AT	1.1	6
26. Titanium	C120AV	1.2	6
27. Aluminum	2017	1.3	11
28. Brass	Casting	1.5	4
29. Pressed carbon	C-13	1.6	8
30. Aluminum	5052	1.7	15
Reference 39.			(U)

Table XIV

CORROSION OF METALS IN LF_2 5-HOUR EXPOSURE AT
-320°F AVERAGE TEMPERATURE* (U)

Material	Type	Corrosion** Rate (mils/year)
1. Nickel	A	24.1
2. Inconel	-	26.7 (U)

Table XIV -- Concluded*(U)

Material	Type	Corrosion** Rate (mils/year)
3. Steel	LC	26.7
4. Tantalum	-	32.4
5. Brass (Amer)	#243	55.6
6. Zircalloy	II	60.4
7. Magnesium	M1A	65.7
8. Nickel	L	71.8
9. Magnesium	HK31A-H24	82.8
10. Ilium	R	87.2
11. Aluminum	1100-H14	87.6
12. Monel	Cast	118.3
13. Zirconium	-	135.8
14. Stainless steel	304	137.6
15. Brass ;	Red	157.7
16. Copper	ETP	157.7
17. Stainless steel	347	157.7
18. Titanium	B120-VCA	157.7
19. Monel	-	160.3
20. Aluminum	2024-T3	166.5
21. Aluminum	5154-H34	192.7
22. Aluminum	3003-H14	210.3
23. Titanium	A-55	232.2
24. Magnesium	AZ81C-T6	577.9

*Based on 2 samples for each material.

**Corrosion rates (expressed in mils/year) based only on 5-hour exposure data, are likely to indicate excessively large corrosion rate values, as initial exposure tends to inhibit subsequent corrosion.

Reference 40. (U)

CORROSION OF MATERIALS BY LIQUID FLUORINE (U)

Material	Type	5-Hour Exposure		24-Hour Exposure		102-Hour Exposure	
		No. of Samples	Corrosion Rate (mils/year)	No. of Samples	Corrosion Rate (mils/year)	No. of Samples	Corrosion Rate (mils/year)
Aluminum	300-H14	5	160.1	2	8.2	2	4.5
Monel	-	4	139.3	3	14.6	2	5.2
Stainless steel	304	4	88.7	3	5.5	2	5.6
Titanium	A-55	2	232.2	1	49.1	1	39.9
Titanium	A-70	2	249.7	2	16.7	-	100.3

Reference 40.

(U)

Table XVI

COMPATIBILITY (U)

Compatibility Classification For Metals				
Corrosion Resistance			Decomposition of Propellant	Shock Sensitivity
Class	Rating	Corrosion Rate (mils./year)		
1	Excellent	1	No	No
2	Good	5	No	No
3	Fair	5 to 50	Some	No
4	Poor	50	Extensive	Yes
The classification of a material is based on the lowest rating of any one of the three properties.				
Compatibility Classification For Nonmetals With A Propellant				
	Class 1	Class 2	Class 3	Class 4
Volume change (percent)	0 to +25	-10 to +25	-10 to +25	<-10 or >+ 25
Durometer reading change	±3	±10	±10	<-10 or >+ 25
Effect on propellant	None	Slight change	Moderate change	Severe
Visual examination	No change	Slight change	Moderate change	Severely blistered, or cracked, dissolved
General usage	Satisfactory, general use	Satisfactory for repeated short term use	Satisfactory for short time use	Unsatisfactory (U)

COMPATIBILITY OF MATERIALS WITH FLUORINE* (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Aluminum 1100	700	600	>600	<673	-320			-310
Aluminum 2017						-320		-310
Aluminum 2024	700	>1,000	1,000	>1,000				-310
Aluminum 3003	700	700	1,000	>1,000		-320		-310
Aluminum 5052						-320		-310
Aluminum 5154	700	>1,000	1,000	>1,000		-320		-310
Aluminum 6061						-320		-310
Aluminum 7079						-320		-310
Eryllium				75				
304 Stainless Steel	400	400	500	>500	-320	-320	-310	
304L Stainless Steel	>70		>406					

*See Table XVI for Code to classification.
Reference 41.

(U)

*See Table XVI for Code to classification.
Reference 41.

Table XVII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
309 Stainless Steel	500			570				
309C _b Stainless Steel	500			570				
310 Stainless Steel	500			660				
316 Stainless Steel	400				-320			
321 Stainless Steel							-310	
347 Stainless Steel	390			500	-310		-320	
410 Stainless Steel				75	-320			
420 Stainless Steel					-320			
430 Stainless Steel		400	390	600				
Carpenter 20	400				-320			
PH15-7 Mo					-320			
AM-350-C					-320			
AM-350-CX					-320			
AM-350-D					-320			(U)

Table XVII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
AM-350-DX					-320			
Cast Iron				75				
Armco Iron	390	500	167					
Silicon Iron				75				
Iron (0.004 SE)		200	400	390				
Iron (0.79 SE)		100	300	390				
Sheet Steel	390	660		500				
SAE 1010	100	200	400	>400				
SAE 1015				570				
SAE 1020				390				
SAE 1030	665		390	500				
Carbon Steel	700			930				
Music Wire				570				
A-Nickel		1,000	750	>1,200				(U)

Table XVII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
D-Nickel		1200	1300	>1300				
L-Nickel		800	1300	>1300			-310	
Nickel (low carbon)	1,100	1200	1300	>1300				
Nickel (electrolytic)	1,100	1200	1300	>1300				
Duranickel	1,100	1200	1300	>1300				
Monel		1000	750	>1200				-310
Cast Monel	400	800	1000	>1000				
K-Monel	600	800	1200	>1200				
Inconel		1000		<750				
Hastelloy B		75	212					
Hastelloy C	95		212					
Hastelloy D			212					
Copper	200	400	800	>800	-320		-310	
Deoxidized Copper				<400				(U)

Table XVII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Copper EPT	200	400	800	>800				
Brass (20 - 30)		200		400				
Brass (red)	200	400	600	>600				-310
Brass (243)	200		500	>500			-310	-310
Brass (low-leaded)								
Brass (yellow)					-320			
Brass (cartridge)					-320			
Brass (casting)						-320		
Bronze	200	400	700					
Copper-Nickel	200	400		>800				
Copper - 10% Nickel					-320			
Copper - 30% Nickel					-320			
Everdur 1010					-320			
Chromium Plate	400							(U)

Table XVII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Lead			100	>100				
Magnesium		500	700					
Magnesium M1A	653	<1,000						
Magnesium AZ81A-T6	691	<1,000						
Magnesium Z81C-T6		400					-310	
Magnesium AZ91C-T6	<1,000							
Magnesium HK-31					-320			
Magnesium HK-31A-H24			1,100				-310	
Magnesium HK-31 (coated with Dow 17)						-320		
Magnesium HM-31					-320			
Magnesium AZ-31					-320			
Magnesium AZ-31 (coated with Dow 17)					-320			
Magnesium MA (1.2% Mn)	140	200						(U)

Table XVII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Magnesium FS-1A	140	200						
Magnesium J-1H	140							
Magnesium M-1A		600	1000				-310	
Magnesium DOW Metal G	570	600						
Molybdenum				75				
Platinum	212							
Gold	212							
Silver	700							
Silver Solder (fluxon)		100						
Tantalum	212		100	>100			-310	
Tin	212		100	>100				
Titanium A-55	212	100		>350				-310
Titanium A-70								-320
Titanium Alloy 6Al-4V		250			-320	-320		-310 (U)

Table XVII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Titanium A-100AT					-320	-320		
Titanium Alloy B-120VCA		350		>350				
Titanium Alloy 16V-2.5 A		300						
Titanium Alloy 8 Mn		300						-310
Titanium 75A								-310
Columbium				75				
Zirconium		100	400	>700				
Zircaloy-2			400	>700			-310	
<u>Inorganic Materials</u>								
Ruby (Al_2O_3)	75	1,400						
Alumina	392	1,400						
Alundum		1,400						
Sintered Corundum		1,400						(U)

Table XVII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Recrystallized Alumina		1400						
Activated Alumina		400		>400				
Soda Glass	212	500		>300				
Pyrex	212	400		>400				
Silica		480		>480				
Transite		100						
Asbestos		100						
Amorphous Carbon		212		>212				
Metal Fluorides		No limit						
CaF ₂ -Na ₂ SiO ₃ Baked	400	400		>400				
Sulfur Cements				>32				
Organic Materials								
Liquids								
KEL F No. 10			<RT					(U)

Table XVII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
N-43 (C ₄ H ₉) 3N			<RT	All temp				
Cenco Hyvac Oil				All temp				
Glyptal				All temp				
Dow Corning Fluid 200				All temp				
Safety Solvent 178				All temp				
Leak Tec				All temp				
Carbon Tetrachloride				All temp				
<u>Greases and Pastes</u>								
KEL F Med. Wax			<RT					
KEL F No. 1 Grease			<KT					(U)

Table XVII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Fluorolube LG and MG			<RT					
Permatex No. 3			<RT					
Q-Seal			<RT					
Blue Goop			<RT					
Molylube			<RT					
Plast-O-Seal			<RT					
Permatex Nos. 1 and 2				All temp				
White Lead				All temp				
<u>Solids</u>								
Teflon	390			>390				
KEL F	RT			>RT				
Neoprene (glass filled)				>RT				(U)

Table XVII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Neoprene	75			All temp				
Rubber				All temp				
GRS Rubber				All temp				
PVC				>RT				
Plexiglas				>RT				
Tygon				>RT				
Pennsalt PCC and PCI				>RT				
Polystyrene				All temp				
Polyethylene				All temp				
Dow Corning Elastomer				All temp				
Molybde No. 2				All temp				
Lytharge and Glycerine				All temp				(U)

Table XVII. Concluded (U;

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Phenolic Plastic (filled)				All temp				
Phenolic Cement				All temp				
Phenol Formaldehyde				75				
Polyfurfuryl Alcohol				All temp				
Polymethyl Methacrylate				All temp				
Polyethane				All temp				
Polyesters				All temp				
Graphite				All temp				
Pressed Carbon C-13	75					-320		(U)

(U) Metals which do not form protective films include silver, cadmium, bismuth, titanium, zirconium, cobalt, tantalum, molybdenum, tungsten, uranium, indium, platinum, and gold.

(U) Water vapor is a prime potential source of trouble; it reacts with fluorine to form a mixture of oxygen and hydrogen fluoride and containing small amounts of ozone, hydrogen peroxide, and oxygen fluoride. Some of the corrosive properties assigned to fluorine by early workers to explain apparatus failures were actually caused by contamination of the gas by hydrogen fluoride.

(U) The fluoride film formed on most metals exposed to F_2 becomes hydrated when exposed to water and turns to a loose powdery substance which easily separtes from the metal. Nickel forms fluoride films which are fairly resistant to hydration although they absorb moisture. Aluminum fluoride films hydrate slowly after an induction period. Copper fluoride films hydrate immediately. Hydrogen fluoride will also disrupt protective fluoride films of some metals by forming bifluoride complexes. Fluorine systems will have to be either kept dry or dried to remove absorbed moisture; the passivity of most metal fluoride films is destroyed by traces of moisture.

(U) Metal oxides may be less reactive to fluorine than their base metals. Time for complete fluorination varies up to many hours, which helps explain variable performance in passivating fluorine systems.

(U) Published articles on materials compatibility with fluorine indicate that nickel and Monel may be used with confidence in liquid fluorine systems. Copper and aluminum 1100, 3003, 2024, and 5052 can be used if the system can be kept dry. The more noble metals can be used at cryogenic conditions, but they fail in gaseous fluorine at elevated temperatures. Other materials can also be used under more restricted conditions. Corrosion of metals forming protective films was generally no more severe in liquid fluorine than in gaseous fluorine. Low carbon steels, which are generally poor both in fluorine resistance and in physical properties at cryogenic temperatures, are to be avoided.

(U) Highly fluorinated compounds such as Kel-F, Viton, and Teflon are the only organic materials possessing some resistance to fluorine. Of these, only Teflon is sufficiently inert for limited structural use. It has been utilized successfully at moderate gas velocities and in liquid fluorine under static conditions. Freedom from contamination is a necessity to prevent secondary reactions which would generate destructive temperatures and initiate a polymer breakdown which forms carbon tetrafluoride.

(U) Nonmetallic materials such as synthetic sapphire, Norbide, and Kintanium are reportedly nonreactive in liquid fluorine. These and other similar materials may be suitable for use in bearings and seals. Among the other oxidation-resistant cermets being considered are ductile magnesium oxide and a titanium carbide-columbium combination.

2. COMPATIBILITY OF MATERIALS OF CONSTRUCTION WITH CHLORINE TRIFLUORIDE

(U) The Pennsalt Chemical Corporation has conducted perhaps the most extensive tests to date on materials compatibility with chlorine trifluoride (CTF or CLF_3) (References 42 and 43). Other companies, including Rocketdyne, have obtained practical experience on the compatibility of actual system components with CTF (Reference 44). The results of the Pennsalt static immersion tests are summarized in Tables XVIII, XIX, and XX. Recommendations by Rocketdyne on compatible materials for specific applications are shown in Table XXI. Table XXII is a compilation of materials which are known to be unsuitable for use in CTF systems. Table XXIII, prepared by the Battelle Memorial Institute, summarizes test results from several researchers on materials compatibility with CTF.

(U) In a search for electrode materials for use in halogen fluoride electrolytes, 19 metals and alloys were tested for compatibility by Douglas (Reference 45). Weighed specimens (coupons approximately 1×1 cm and of different thicknesses ranging from 3 to 60 mils, depending on the sheet stock available) were exposed to liquid bromine trifluoride at 25°C and liquid chlorine trifluoride near the boiling point (11.8°C) for periods up to 24 hours.

(U) Qualitative observations are given in Table XXIV. Many of the metals tested either ignite violently or dissolve rapidly in both chlorine and bromine trifluorides. Aluminum, cadmium, copper, lead, Monel, and nickel suffer only negligible to moderate weight changes and some tarnishing in 24-hour exposure. Magnesium, silver, and zinc are only slightly tarnished in chlorine trifluoride, but are extensively corroded by bromine trifluoride. Gold dissolves fairly slowly in bromine trifluoride, but was not tested in chlorine trifluoride. Platinum is the only metal tested which does not change weight or appearance in 24-hour exposure.

(U) The Pennsalt researchers (Reference 43) subjected aluminum (1100, 2014 T6, and 6061 T6), nickel 200, 347 SS, and 410 SS, and Teflon to a dynamic flow test of CTF through an orifice in the specimen at approximately 190 ft/sec for approximately 1 hour. No erosion of the orifice was noted on the metal specimens. However, the Teflon orifice was seriously eroded in less than 30 min.

(U) In an ultra high velocity, high-temperature impingement test was conducted in which a small amount of steel wool was placed in the path of the jet immediately prior to impingement on the $3/8$ -in.-thick specimen, to ensure ignition of the specimen. Although this is not a realistic test for evaluating materials for an oxidizer system, it does provide information on the relative resistance of the materials tested at high temperature and flow conditions. If the volume of metal lost during the test is assumed to be the criterion, the order of decreasing resistance of the metals tested is: copper, nickel, Monel, 347 SS, 304 SS,

Table XVIII
CORROSION RATES IN 21-DAY IMMERSION TESTS IN
LIQUID ClF_3 AT 30°C^* (U)

Material	Alloy	Hours of Exposure	Average Corrosion Rate (mils/year)
1. Nickel	A	522	0.00
2. Inconel	-	522	0.00
3. Aluminum	1061	524	0.01
4. Aluminum	1100	524	0.01
5. Aluminum	2024	524	0.01
6. Aluminum	3003	524	0.01
7. Aluminum	5052	524	0.01
8. Aluminum	7079	524	0.01
9. Monel	-	522	0.01
10. Incol		522	0.01
11. Stainless steel	PH15-7Mo (Cond. RH 950)	522	0.01
12. Stainless steel	PH15-7Mo	522	0.01
13. Low Carbon steel	1010	523	0.01
14. Low Carbon steel	1010 (Coated with Fosbond 27)	523	0.01
15. Stainless steel	304	383	0.02
16. Stainless steel	Carpenter #20-Cb	383	0.02
17. Aluminum bronze, 8%	Ampco 8	520	0.03
18. Brass	yellow	524	0.03
19. Nickel silver, 18%	Alloy A	524	0.03
20. Cupro-Nickel, 30%	-	524	0.03
21. Magnesium	AZ31B	520	0.03
22. Stainless steel	304	568	0.03
23. Stainless steel	316	383	0.03
24. Stainless steel	403	383	0.04
25. Low Carbon steel	1010 (Coated with Fosbond 27)	523	0.04

(U)

Table XVIII. Concluded (U)

Material	Alloy	Hours of Exposure	Average Corrosion Rate (mils/year)
26. Stainless steel	347	383	0.05
27. Stainless steel	Carpenter #20-Cb	568	0.05
28. Magnesium	HM21A	526	0.05
29. Stainless steel	316	568	0.06
30. Brass	Rule	524	0.07
31. Stainless steel	403	568	0.07
32. Stainless steel	347	568	0.09
33. Copper	ETP	520	0.11
34. Beryllium copper, 2%	Berylco 25	520	0.11
35. Copper	DHP	520	0.12
36. Magnesium	AZ31B	520	0.13
37. Phosphor bronze, 5%	Grade A	520	0.15
38. Teflon	CaF ₂ -filled	520	0.16% wt gain
39. Teflon	-	520	0.70% wt gain
40. Kel-F	-	520	1.02% wt gain

*Based on two specimens per each material.

Reference 42. (U)

1020 carbon steel, PH15-7Mo, 410SS, 6061 aluminum, 1100 aluminum, and 2024 aluminum. There was little difference between copper and nickel; both were resistant to attack under the conditions used. Carbon steel 1020, 410 stainless steel, and PH15-7Mo were all about equally attacked. The aluminum targets were the only ones that were cut through.

(U) The following useful information was obtained during the dynamic flow testing program:

- a. An air-operated stainless steel valve functioned satisfactorily with oxidizer at pressures up to 600 psig. The oxidizer-wetted parts of the valve were 347 stainless steel bellows and 316 stainless steel valve seat and valve body.

Table XIX
CORROSION RATES IN 21-DAY IMMERSION TESTS
IN Cl F₃ VAPOR AT 30°* (U)

Material	Alloy	Hours of Exposure	Average Corrosion Rate (mils/year)
1. Aluminum	1100	501	0.02
2. Monel	-	501	0.02
3. Nickel	A	501	0.06
4. Low-Carbon steel	1010	501	0.07
5. Stainless steel	316	501	0.09
6. Copper	ETP	501	0.11
7. Stainless steel	403	501	0.13
8. Aluminum bronze 8%	(Ampco 8)	501	0.15
9. Magnesium	AZ31B	501	0.15
10. Brass	Yellow	501	0.60
*Based on two specimens for each material. Reference 42. <div style="text-align: right;">(U)</div>			

- b. Silver solder proved very satisfactory for sealing all of the stainless steel threaded pipe joints.
- c. Inert arc-welded pipe connections proved completely trouble free.
- d. Metal valves were cleaned as for oxygen service; and Kel-F oil was used as lubricant on all moving parts except the valve seat.
- e. The small amount of fine metal chips from the pipe threading operation that was introduced into the system was successfully removed by a stainless steel filter. These fine metal chips did not react with the oxidizers.

(U) The following pertinent conclusions were reached as a result of the test program:

- a. Weldments of the materials tested had corrosion resistance equal to the base material.

Table XX
CORROSION RATES OF METALS IN CHLORINE TRIFLUORIDE* (U)

Material	Alloy	Average Corrosion Rate (mils/year)		
		21-Day Exposure at 300C	21-Day Exposure at 660C	1-Year Exposure at 300C
1. Aluminum	1100-0	0.00	0.01	0.00
2. Aluminum	2024-T6	0.00	0.01	0.00
3. Aluminum	6061-T6	0.00	0.01	0.00
4. Monel	400, annealed	0.00	0.01	0.00
5. Nickel	200, annealed	0.00	0.01	0.00
6. Stainless steel	PH15-7Mo (RH-1050)	0.01	0.07	0.01
7. Stainless steel	AM-350, (SCT-850)	0.01	0.05	0.00
8. Stainless steel	347, annealed	0.01	0.07	0.00
9. Stainless steel	410, hard and tempered	0.03	1.83	3.08
*Based on 12 specimens of each material; 4 each exposed to liquid, vapor, and interface between liquid and vapor. Reference 43.				
(U)				

Table XXI
MATERIALS COMPATIBLE WITH CHLORINE TRIFLUORIDE (U)

Structural Application	Liquid CTF	Gaseous CTF
1. Exhaust gas neutralizer and/or burnoff stacks	-	Mild steel
2. Gaskets	Aluminum 1100 Lead indium alloy Copper Copper laminated Stainless steel 303 Sterling silver	Aluminum 1100 Lead indium alloy Copper Teflon Tin
3. Lines and fittings	300 Series Stainless steels Nickel Monel Inconel Aluminum 1160 Aluminum 6063 Aluminum 3003 Aluminum 2024 Aluminum 5052 Aluminum 6061 Aluminum 6066 Copper	Same as for liquid
4. Lubricants	None	None
5. Orifice - meter	K-Monel	None
6. Pump materials (volute, impeller, and shaft)	Aluminum 356 Aluminum tens 50 Aluminum 6061 Aluminum 6066 Rene 41 Tin Carbon Monel	Aluminum Aluminum tens 50 Tin Boron carbide
Reference 44. (U)		

Table XXI -- Continued (U)

Structural Application	Liquid CTF	Gaseous CTF
7. Rotating seals	Tin indium alloy Copper Indium Silver solder Boron carbide Carbon	Tin indium alloy Copper Indium Silver solder Boron carbide Carbon
8. Wear plates	Rene 41 Stainless steel Stainless steel-347 Nitalloy Chromium plated-steel	Rene 41 Stainless steel Stainless steel-347 Nitalloy Chromium plated-steel
9. Bearings	Aluminum Copper Bronze	Aluminum Copper Bronze
10. Valve bellows (packless valves)	Stainless steel 321 Stainless steel 347 Monel Aluminum	Stainless steel 321 Stainless steel 347 Monel Aluminum
11. Valve bodies	Most 18-8 stainless-steels Bronze K-Monel Aluminum casting alloys Aluminum tens 50 Aluminum 356	Most 18-8 stainless-steels Bronze K-Monel Aluminum casting alloys Aluminum tens 50 Aluminum 356
12. Valve packing	Teflon-Chevron	Teflon-Chevron Copper-laminated Teflon (U)

Table XXI -- Concluded (U)

Structural Application	Liquid CTF	Gaseous CTF
13. Valve plugs	Stainless steel 304 Stainless steel 321 Stainless steel 347 K-Monel Inconel	Stainless steel 304 Stainless steel 321 Stainless steel 347 K-Monel Inconel
14. Valve seats	Copper Aluminum 1100	Copper Aluminum 1100
15. Tanks	Same materials as for lines and fittings	Same materials as for lines and fittings
<p>NOTE: CTF will occasionally ignite fluorocarbon polymers, therefore each Teflon gasket should be exposed to CTF prior to its use to prove compatibility. No point on the exposed Teflon gasket or sealing surface should be more than 0.002 or 0.003 in. removed from a metal heat conductor. CTF does not react with Pyrex glass, but will attack it via HF upon exposure to moisture.</p> <p style="text-align: right;">(U)</p>		

Table XXII

MATERIALS UNSUITABLE FOR USE IN CTF SYSTEMS

1. Titanium	9. Graphite with plastic binders
2. Columbium	10. Carbon with plastic binders
3. Molybdenum	11. Glass and glassed steel
4. Polyethylene	12. Silicones and fluorosilicones
5. Saran	13. Hydrocarbon or ester base lubricant
6. Nylon	14. Koroseal
7. Rubber (buna and butyl)	15. Viton-A
8. Neoprene	16. Glytal sealants
	17. Epoxy sealants
Reference 44.	
(U)	

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Metals								
Aluminum 1060					85			
Aluminum 1100	85		644		85			
Aluminum 1100 welded	85				85			
Aluminum 2014 welded	85				85			
Aluminum 2024					85			
Aluminum 3003					85			
Aluminum 5052					85			
Aluminum 6061	85				85			
Aluminum 7079					85			

*See Table XVI for Code to Classification.

Reference 42.

(U)

***See Table XVI for Code to Classification.**

Reference 42.

(U)

Table XXIII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Metals								-105
Columbium								
Copper, ETP	85		590		85			
Copper, DHP					85			
Beryllium Copper, 2%					85			
Phosphor Bronze, 5%					85			
Aluminum Bronze, 8%	85				85			
Yellow Brass	85				85			
Red Brass					85			
Tin Bronze	75							
1010 Steel	85		685		85			
1010 Steel (coated with Fosbond 40)					85			
1010 Steel (coated with Fosbond 27)					85			(U)

Table XXIII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Metals								
403 Stainless Steel	85				85			
304 Stainless Steel					85			
316 Stainless Steel	85				85			
347 Stainless Steel					85			
AM 350						75		
A-286						75		
Carpenter 20					85	75		
PH 15-7Mo (RH 950)					85			
PH 15-7Mo (TH 1050)					85			
410 Stainless Steel (welded)	85				85			
347 Stainless Steel (welded)	85				85			
AM-350 (welded)	85				85			
PH 17-7Mo (welded)	85				85			(U)

Table XXIII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Metals								
Lead		75						
Magnesium, AZ-31B	85				85			
Magnesium, HM-21A					85			
Magnesium, HK-31A					85			
A-Nickel	85				85			
Monel	85				85			
Inconel					85			
Inconel X							75	
Incoloy					85			
Hastelloy X							75	
Rene 41						75		
Cupro-Nickel, 30%					85			
Nickel 200 (welded)	85				85			(U)

Table XXIII. Continued (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Metals								
Monel 400 (welded)	85				85			
Nickel-Silver, 18% (Alloy A)					85			
Thorium		660						
Titanium, 100A				75				75
Titanium, C-120AV								75
Titanium, A-110 AT								75
Uranium				400				
Zirconium				645				
Nonmetals								
Glass	75							
Pyrex Glass		75						
Stoneware	75							(U)

Table XXIII. Concluded (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Nonmetals								
Neoprent Rubber	75							75
Carbon, Karbate No. 15								75
Graphite, Karbate No. 25						75		
Graphite Graphitar 39								75
Graphitar 67								75
Graphite AUT-72								
Teflon	75				85			
KEL-F		75				85		
Polyvinylidene Fluoride (RC-2525)					85			135
								(U)

Table XXIV
QUALITATIVE METAL REACTIONS WITH CHLORINE
AND BROMINE TRIFLUORIDES (U)

Metal or Alloy	Halogen* Fluoride	Wt. Change of Metal After 24-hour Exposure	Remarks
Al	$\text{ClF}_3(1)**$	Unchanged	very slightly tarnished
	$\text{BrF}_3(1)$	Slight loss	Gray deposits
Sb	$\text{ClF}_3(1)$	Destroyed	Ignited — reaction violent
	$\text{BrF}_3(1)$	Destroyed	Ignited — reaction violent
Cd	$\text{ClF}_3(1)$	Slight loss	Tarnished
	$\text{BrF}_3(1)$	Moderate loss	Tarnished
Cu	$\text{ClF}_3(1)$	Slight loss	Tarnished
	$\text{BrF}_3(1)$	Moderate gain	Tarnished
Au	$\text{BrF}_3(1)$	Destroyed	Tarnished before dissolving — mild reaction
Pb	$\text{BrF}_3(1)$	Moderate loss	Tarnished
Mg	$\text{ClF}_3(1)$	Slight gain	Tarnished, metal floated
	$\text{BrF}_3(1)$	Heavy loss	Gray deposits, metal floated
Mo	$\text{ClF}_3(1)$	Destroyed	Ignited — violent reaction
	$\text{BrF}_3(1)$	Destroyed	Delayed ignition while dissolving — violent reaction
Monel	$\text{BrF}_3(1)$	Unchanged	Slightly tarnished
<p>*Test temperature for BrF_3 is at room temperature and for ClF_3 is near its boiling point (b. p. 11.8°C).</p> <p>** (1) denotes liquid phase.</p> <p style="text-align: right;">(U)</p>			

Table XXIV. Concluded (U)

Metal or Alloy	Halogen* Fluoride	Wt. Change of Metal After 24-hour Exposure	Remarks
Ni	ClF ₃ (l) BrF ₃ (l)	Unchanged Moderate loss	Slightly tarnished Deposits
Nb	BrF ₃ (l)	Destroyed	Ignited—violent reaction
Pt	ClF ₃ (l) BrF ₃ (l)	Unchanged Unchanged	No detectable change No detectable change
Ag	ClF ₃ (l) BrF ₃ (l)	Moderate loss Destroyed	Tarnished Tarnished before dissolving—mild reaction
Ta	ClF ₃ (g)** BrF ₃ (l)	Destroyed Destroyed	Ignited—violent reaction Ignited—violent reaction
Sn	BrF ₃ (l)	Not measured	Vigorous bubbling—violent reaction
Ti	ClF ₃ (l) BrF ₃ (l)	Destroyed Destroyed	Vigorous bubbling—violent reaction Ignited—violent reaction
Ti Alloy	ClF ₃ (l)	Destroyed	Vigorous bubbling—violent reaction
(5Al, 2.5Sn)	BrF ₃ (l)	Destroyed	No ignition—moderate bubbling
Ti Alloy	ClF ₃ (l)	Destroyed	Vigorous bubbling
(5Al, 4V)	BrF ₃ (l)	Destroyed	Ignited—violent reaction
Zn	ClF ₃ (l) BrF ₃ (l)	Slight gain Heavy loss	Tarnished Tarnished
*Test temperature for BrF ₃ is at room temperature and for ClF ₃ is near its boiling point (b.p. 11.8°C). **(g) denotes gas phase.			

- b. Typical alloys of aluminum, copper, magnesium, nickel, low carbon steel, and stainless steel were found to be highly resistant to CTF at 30°C.
- c. Titanium, columbium, molybdenum, carbon, and graphite are rapidly attacked by CTF.
- d. Teflon and Kel-F can be used with CTF, but only with extreme care and only under mild conditions of heat and shock. Both plastics adsorb moderate amounts of oxidizer and may undergo structural changes. Occasionally they do fail, but the failure is not likely to spread to adjoining metal. These plastics are best used in vapor and when provided with good heat release. They should never be used in service with this oxidizer when subjected to heat, shock, or flow conditions.
- e. At 66°C, type 410 stainless steel had a marked increase in corrosion compared to the results at 30°C (see Table XX). The specimens had a more voluminous corrosion deposit, part of which was powdery and loosely held. Insoluble corrosion products of this type, carried along in the liquid stream, could prove troublesome at flow restrictions such as valves. Therefore, the use of the straight chromium stainless steels in this oxidizer is not recommended for conditions exceeding ambient temperature. It would appear that a minimum nickel content in combination with the chromium is necessary for corrosion resistance above ambient temperature.
- f. The results of dynamic testing of the metals exposed to CTF was favorable for its safe practical use. CTF is not shock-sensitive in contact with aluminum, nickel, or stainless steel, and it does not show an erosive attack on the common metals at normal temperatures and flow rates. Nickel or high nickel alloys should be used where extreme conditions of temperatures or flow are expected.
- g. Because of the possible selective attack of CTF on columbium and molybdenum, light structural elements such as diaphragms, bellows, and fine wire made from certain stainless steels containing these stabilizing elements in significant amounts should not be used. In such elements, a small surface corrosion could result in a serious change in the mechanical properties of the object.

(U) An observation was presented that only those metals that form volatile fluorides or fluorides soluble in CTF have high corrosion rates at low temperatures. It was noted that iron, copper, nickel, aluminum, and magnesium all had very low corrosion rates and that the fluorides of these metals have low vapor pressures up to several hundred degrees. Titanium, molybdenum, uranium, and columbium all form fluorides that have high vapor pressures at atmospheric temperatures or form complex ions that are soluble in CTF. All of these materials show high attack in liquid CTF at low temperatures.

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(U) It was noted by Rocketdyne (Reference 44) that fluoride deposits accumulate on stainless steel components after prolonged exposure with CTF. The corrosion product consists primarily of hydrolyzed fluoride salts of iron, chromium, and nickel. Of the 300 series stainless steels, 304 gives superior service. Types 303, 321, and 347 stainless steels have greater corrosion buildup because of the reaction with impurities that are trapped in inclusions in these steels. The presence of moisture in a CTF system accelerates corrosion (hydrolyzed fluoride salts), and will cause embrittlement of the protective fluoride film. Mechanical shock can then cause separation of this coating and expose unpassivated surfaces to CTF. Clad steels are unreliable for use with CTF because of difficulty in ensuring continuity and quality of the covering material.

3. COMPATIBILITY OF MATERIALS WITH FLOX

(U) Because FLOX is a mixture of fluorine and oxygen and fluorine is more reactive than oxygen with materials of construction, it may be expected that materials which are compatible with fluorine will also be compatible with FLOX. Table XXV, compiled by the Battelle Memorial Institute, is a summary of the test results from FLOX compatibility testing.

4. COMPATIBILITY OF MATERIALS WITH COMPOUND A

(C) It appears that only a limited amount of data on material compatibility with Compound A has been published. The following data was published by Rocketdyne (Reference 46).

(U) Materials compatible with Compound A are:

Magnesium	Chromium	Nickel	Palladium	Gold
Aluminum	Manganese	Copper	Cadmium	Nichrome V
Indium	Cobalt	Zinc	Platinum	

(U) Pure nickel and Hastelloy C were the most corrosion resistant metals.

(U) Kel-F and Teflon were used under limited and controlled service conditions.

(U) Metals which are reactive at 250°C are as follows:

Lithium	Niobium	Mercury	Arsenic
Titanium	Molybdenum	Boron	Antimony
Vanadium	Tungsten	Silicon	

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COMPATIBILITY OF MATERIALS WITH FLOX* (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
FLOX 40 (40% F ₂ - 60% O ₂)								
Aluminum, 2014 T6					-320			
Aluminum, 5052-H34					-320			
Aluminum, 6061-T6					-320			
4340 Steel					-320			
301 Stainless Steel					-320			
304 Stainless Steel					-320			
347 Stainless Steel					-320			
Monel					-320			
Incone X					-320			
Teflon TFE	RT							

*See Table XVI for code to classification.
Reference 41.

*See Table XVI for code to classification.

Reference 41.

(5)

Table XXV. Concluded (U)

Material	Temperature °F							
	Gas				Liquid			
	Class 1	Class 2	Class 3	Class 4	Class 1	Class 2	Class 3	Class 4
Teflon FEP	RT							
KEL-F 81	RT							
Armalon PDX 7700				RT				
Asbestos-Stainless Steel Gasket				RT				
Oxylube 701 on 347 SS						-320		
FLOX-20 (20% F2 - 80% O2)								
Aluminum, 2014					-320			
Cad Plated 4037 Steel					-320			
Oxylube 701 on 347 SS						-320		
								(U)

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(U) In general, materials which are compatible with CTF are compatible with Compound A.

5. MISCELLANEOUS COMPATIBILITY DATA

(C) The following comments were furnished by Rocketdyne (Reference 47) and are based on their experience with compatibility of materials with the fluorine oxidizers. They have found that contamination from water is the biggest problem. The fluoride film formed on most metals exposed to a fluorine oxidizer becomes hydrated when exposed to water and turns to a loose powdery substance which will break away and contaminate the system during a subsequent flow of oxidizer. Therefore, when a line is disconnected, great care must be taken to prevent exposure to moist air. Copper and most nickel alloys, including Monel, are subject to this type of contamination. Rocketdyne has found pure nickel to be relatively free of this problem and Hastelloy C to be the most resistant of the nickel alloys.

(C) Rocketdyne tested Lucalox, an aluminum oxide material produced by General Electric, with Compound A and found essentially no reaction. They also commented that titanium carbide has an interesting reaction when exposed to fluorine. It has a very slow reaction which produces a liquid lubricant and therefore has possibilities for application where friction is a consideration.

Appendix IV

FACILITY, RANGE, AND REGULATORY AGENCY CONTROLS

1. TOXIC LIMIT DATA

(U) Maximum allowable concentrations (MAC) or (more recently) threshold limit values (TLV) adopted by the American Conference of Governmental and Industrial Hygienists (ACGIH) for fluorine and CTF are fixed at the same value, 0.1 ppm by volume (Reference 48). A sample graphical solution of the equation for fluorine is shown in Figure 41. TLV's are based on the best available information from industrial experience, experimental animal studies, experimental human studies, and a combination of all three, when possible. As such, they are intended to be used as guides of time-weighted average concentrations to which most workers could be exposed for 8 hours each day, 5 days per week, for life without adverse effect.

(U) Following World War II, the production and use of large quantities of fluorine by the Atomic Energy Commission (AEC) and the chemical industry, and its planned use for rocket propulsion prompted more extensive toxicological investigations to determine realistic short-term exposure values applicable to operational personnel. Some toxicity studies on hydrogen fluoride (HF) were completed in 1934 and 1935 by W. Machle, et al. (References 49 and 50). HF was a well known and much-used chemical, whereas fluorine usage was slight, which may explain the dearth of toxicological data available up to 1945. Additional work was reported on fluorine (F₂) in that year by N. Eriksen, H. Stokinger, et al (References 51 through 53), with F₂ and HF data by Voegtlin and Hodge (Reference 54) and later work on HF by Carson, et al in 1961 and 1963 (References 55 and 56). A distinct difference in chronic toxicity between F₂ and HF has been noted, but for short exposures, both exhibit fast-acting irritant action on the eyes and respiratory mucosa. With high concentrations, exposed personnel would be forced to vacate the area because of involuntary choking or spasm.

(U) One manufacturer of fluorine has found that 25 to 35 ppm F₂ forces the vacating of an area (Reference 57).

(U) Fluorine is easily detectable by odor, and personnel are trained to leave an area in a few minutes if the odor is detected. However, odor detection is of limited value, in that it is impossible to determine concentrations quantitatively.

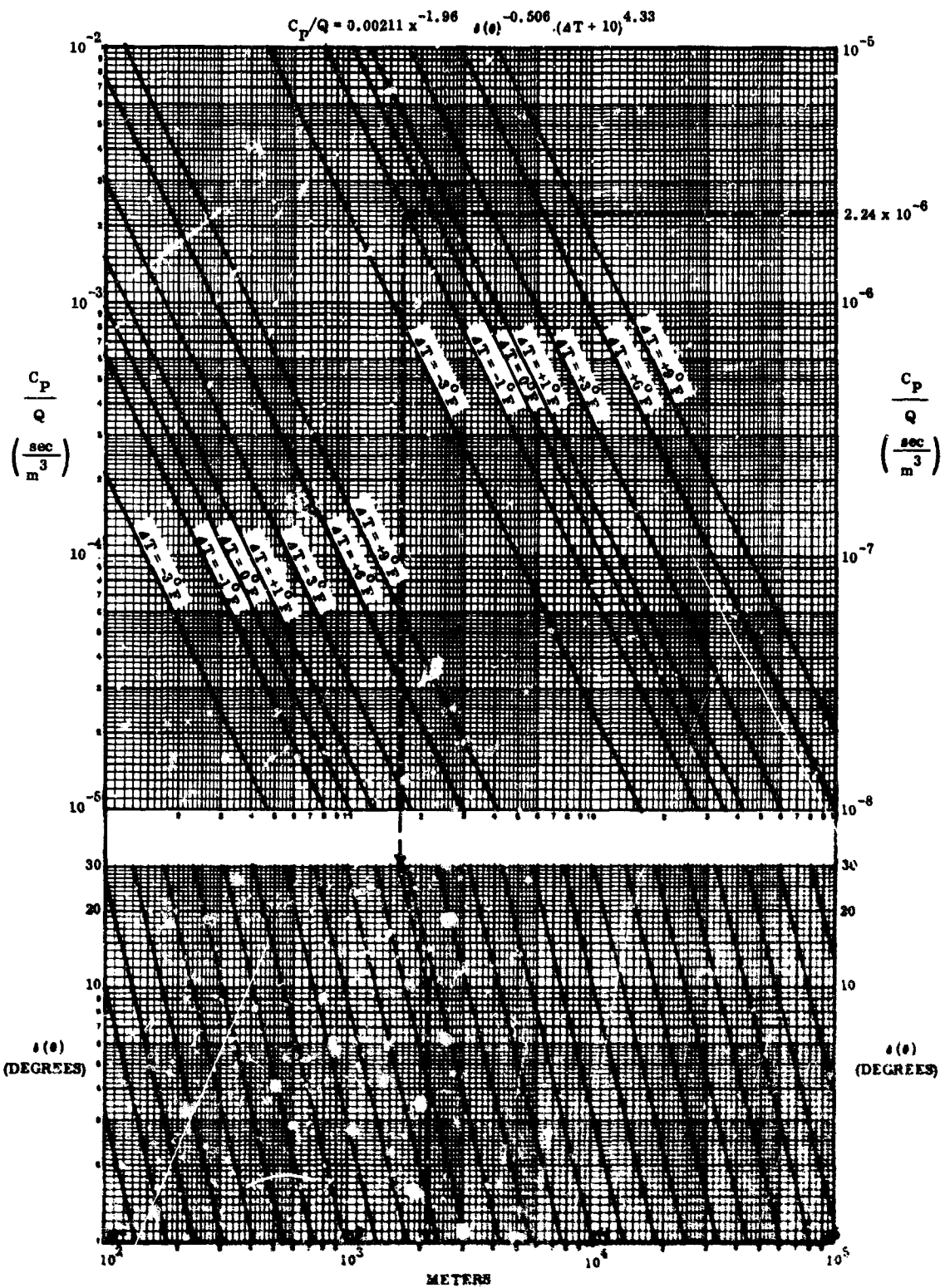


Figure 41. Graphical Solution of WINDS Prediction Equation

(U) The amounts by which the concentrations may exceed the TLV recommended values (F₂-0.1 ppm, HF-3 ppm) up to the intolerable concentrational values for short periods without injury to health depends on a number of factors:

- a. Whether high concentrations even for short exposures produce acute poisoning.
- b. Whether the effects are cumulative.
- c. The frequency and duration of the exposures.

(U) For fast-acting substances, a more appropriate limit, known as a ceiling (c) limit, was agreed to be more applicable than the TLV. Certain ceiling limits have been agreed to and adopted and published by the ACGIH 26th Annual Meeting, April 1964. In general, the bases for assigning a (c) value rest on whether exposure to concentrations above a proposed limit for periods up to 15 min. may result in:

- a. Intolerable irritation.
- b. Chronic or irreversible tissue change.
- c. Narcosis of sufficient degree to increase proneness to accident, impair self-rescue, or materially reduce work efficiency.

(U) In addition to these values, the ACGIH, in conjunction with the Advisory Center on Toxicology of the National Academy of Sciences (NAS) National Research Council (NRC) have recommended emergency tolerance limits (ETL) and emergency exposure limits (EEL) for fluorine oxidizers (Reference 58). The limits are set to give guidance in the control of single, short-duration exposures of personnel to toxicants released to atmosphere.

(U) ETL and EEL values differ only in the degree of hazard risk encountered operationally. ETL's are guideline levels for control of predictable short-duration occupational exposures that may be repeated with competent medical control. On the other hand, EEL's are guideline levels for control of short-duration occupational exposures of the rare, but unpredictable variety. EEL values are concentrations which the normal individual can tolerate without permanent adverse affects, but not necessarily to the exclusion of irritation or mild discomfort. EEL's are based on the following:

- a. Exposures at these levels will be from unplanned accidents, not the result of engineering controls designed to hold exposures at these levels.
- b. Normally prevailing values of airborne contamination in the occupational environment will be below TLV for continuous exposure and below ETL for short-time exposures.

- c. Normally prevailing values of airborne contamination for the nonoccupational, off-base environment will be below standards of good community atmospheric pollution control.
- d. These accidental exposures will be single events; that is, if a man were exposed at these levels, further exposure would be prevented until he regains his normal resistance.
- e. Men who could be exposed under these conditions will not be hypersensitive to or predisposed to disease from the specific contaminant.
- f. Persons who could be exposed under such conditions would be under medical surveillance.
- g. The probable severity of injury from secondary accidents, resulting from impairment of judgment and coordination, will be considered in applying these values. A degree of temporary intoxication that prevents self-rescue is not considered acceptable.
- h. Safety factors such as those used in deriving TLV's and air-quality guides are not applied to EEL's except in cases of low confidence in the extrapolation of animal data to man.
- i. Mixtures of contaminants that may produce possible interactions, such as potentiation or antagonism, have not been considered in proposing EEL values.
- j. EEL's are intended to provide a finite number which could be introduced into atmospheric diffusion equations to predict downwind vapor hazards for the purpose of siting facilities within a Government reservation.

(U) Because the risks attendant upon the use of EEL criteria are great, an intensive effort to define more certainly fluorine-containing oxidizer hazards is currently underway, funded by the NASA and the Air Force. Results from these study programs will probably not become available in full force until 1966 or 1967 (Reference 59).

(U) Recent contacts with personnel of the Aeromedical Research and Development Laboratory, Toxic Hazards Branch (Reference 60) indicate that preliminary results from current animal toxicity work with fluorine are not expected to change the recommended EEL's promulgated by NAS-NRC.

(U) A summary of the toxic hazard concentrational limits of fluorine and fluorine containing oxidizers appears in Table XXVI. The lethal concentration (LC 50) values are preliminary average figures reported by P. Ricca, KSC Safety Center. This work, funded by NASA, done by M. Keplinger at the University of Miami, is progressing and is expected to yield gross pathological and toxic effects data on rats, mice, guinea pigs, and rabbits exposed to lethal concentrations of fluorine.

Table XXVI

INHALATION LIMITS AND TOXICITY OF FLUORINE
CONTAINING PROPELLANTS (U)

Propellant	TLV		EEL						LC 50	
			Occupational		Community					
	PPM (Vol)	Time	PPM (Vol)	Time (min.)	PPM (Vol)	Time (min.)	PPM (Vol)	Time (min.)	PPM (Vol)	Time (min.)
Fluorine F ₂	0.1	8 hr	3	10	0.5	10	~700	5		
			2	30			~370	15		
			1	60			~270	30		
Hydrogen fluoride HF	3	8 hr	20	10	5	10	4060	5		
			10	30			~2600	15		
			8	60			~1800	30		
Chlorine trifluoride CTF	0.1	8 hr	7	10						
			3	30						
			2	60						
Oxygen difluoride	0.05	8 hr	0.5	10						
			0.2	30						
			0.1	60						
Compound A*	0.1*	8 hr*								

*TLV not officially established, but will probably be the same as for F₂ and CTF. EEL and LC 50 values have not been determined as of January 1966 (Reference 60).

TLV -- Threshold Limit Value.
EEL -- Emergency Exposure Limit.
LC 50 -- Lethal Concentration (50% deaths in test animals).

(U)

*TLV not officially established, but will probably be the same as for F₂ and CTF. EEL and LC 50 values have not been determined as of January 1966 (Reference 60).

TLV -- Threshold Limit Value.

EEL -- Emergency Exposure Limit.

LC 50 -- Lethal Concentration (50% deaths in test animals).

(U)

(U) H. F. Smyth, Jr. and coworkers (Reference 58) pointed out that there is no justification for distinguishing between ETL's and EEL's, since it is recognized that both occupational and nonoccupational exposures can occur at predictable time intervals. Repeat exposures to EEL concentrations will be permitted only with medical monitoring and authorization.

(U) Safety factors, often used in arriving at TLV's, are not applied to the EEL except in cases of low confidence in the extrapolation to man of animal-derived data (usually LC 50 values). TLV safety factors often are established at levels of 10x or more, depending on the degree of industrial experience and known pharmacology of the toxicant.

(U) Because EEL's are established for cases in which personnel may be exposed to higher concentrations for short periods of time, they are intended as guides for the specialist in range safety and toxicology, to aid him in making realistic judgments which will give maximum protection consistent with efficient launch site operations.

(U) Employed by the specialist in this way, EEL's are used as peak values that should not be exceeded except in emergency cases where health risks are justifiable in order to prevent a still more serious event or in the advance planning for particular launch or test site emergencies. The use of these limits as time-line exposures, discriminating between dangerous and tolerable exposures, is not implied, and should be avoided. Extrapolations of these limits to other exposure periods by means of calculations adjusted from concentration-time product or total integrated doses (TID) are not necessarily valid. For example, the 1966 peak concentration limit recommended by NAS-NRC for 10-min. exposure to fluorine is 3 ppm. The TID in this case equals 30 ppm-min. Using this value to calculate a peak concentration allowable for 0.5 min. exposure (30 ppm-min. / 0.5 min.) yields 60 ppm, a peak concentration which is intolerable even for 0.5 min. Thus, it is clear that peak concentrations are as important as exposure time in judging the degree of hazard. However, total integrated doses based on peak concentration x time are more valid in terms of the toxic hazard if the concentration does not exceed intolerable levels; e. g., 10 ppm-min. dose is equivalent to 1 ppm-10 min.

(U) The emergency exposure limits imposed by cognizant authorities for control at a number of sites after careful evaluation show a considerable spread in permissiveness. During the past 3 years, special sets of EEL's, ETL's, TLV's, and total or average dosage values have been established for sites under contract to do fluorine R&D for the Air Force and NASA. These include those set by the USAF Surgeon's Office, SSD, Los Angeles, for Rocketdyne and TRW (Reference 61) for specific use at rocket engine test sites on the west coast. Other facilities include those sited under NASA contracts for GD/A at Sycamore Canyon and Douglas at Edwards AFB (A-23 location) (Reference 62). Table XXVII summarizes the specific site deviations permitted or recommended by various cognizant authorities.

Table XXVII
HISTORICAL SUMMARY OF TOXIC LIMITS IMPOSED FOR USE OF F2
AT ROCKET TEST FACILITIES, 1963 TO 1966 (U)

Testant, Facility, and Date	Exposure Time (min.)	Conc. in Air (ppm) (%V)	TLV (ppm-min.)	Toxicity Population Category	Expected Effect on Humans	Reference and/or Authority
LP2 NASA-Lewis and others Aug. 1963 (10)	5	2.5	10	ETL's	No pathological changes or sen- sory discomfort	NAS-NRC Advisory Center on Toxicology
	15	1.0	23	(rare single events)		
	30	1.0	30			
	60	0.5	30	ETL on-site TLV off-site	No pathology changes Detected by odor	NAS-NRC
LP2 NASA sites incl. RSC June 1964	5	2.5	12.5	ETL's for Group I - controlled personnel	Discomfort with no perma- nent health impairment (single event)	ACGIH (includes 2x safety factor due to lack of data)
	15	1.0	22.5			
	30	1.0	300			
	60	0.5	300	ibid	ibid	
LP2 NASA sites incl. RSC June 1964	5	5	2.5	ETL's for Group II uncontrolled community	No health impairment, slight discomfort	ACGIH
	15	3	4.5			
	30	2	50			
	60	1	60	ibid	ibid	
LP2 NASA sites incl. RSC June 1964	5	5	150			
	15	1.5	22.5			
	30	1.0	300			
	60	0.5	300			
LP2 Rocketdyne GDA, DAC Nov. 1964	5	5	2.5	ETL (ETL) on-site exposures recovery from repeat exposures implied	No impairment of health, some discomfort	NAS-NRC committee of toxicol- ogy meet- ing, NASA Safety Director, and USAF
	15	3	4.5			
	30	2	60			
	60	1	60			
LP2 Rocketdyne GDA, DAC Nov. 1964	5	10	150			
	15	3.0	300			
	30	1.0	300			
	60	0.5	400			
LP2 NASA-Lewis March 1965	5	2.5	10-25	ETL's	No health impairment, mild discomfort	Suggest to NAS- NRC by NASA- Lewis (U)
	15	1.0	23-45			
	30	1.0-2	30-60			
	60	0.5-1	30-60			

(U) Several of the assumptions listed as basic conditions applicable to the use of EEL values for control of personnel subjected to short-term inhalation of fluorine oxidizers serve to emphasize certain vague informational areas. Much animal toxicity research presently underway must be completed to define the limits. Furthermore, in the case of fluorine, the toxicity situation is continuously changing during emissions into air because of reaction with water vapor to form HF. The rate of conversion is not accurately known and a more or less subjective approach must therefore include EEL's for both substances

(U) The primary effects of short-duration exposures to F_2 and HF at levels above the ETL are caused by their irritant actions on the tissues of the eye and respiratory tract tissues. P. Ricca (Reference 63) points out that industrial and animal studies demonstrate that acute exposures cause pathological lung changes long before the occurrence of significant biochemical, hematological, weight, or skeletal changes. Reference 63 also shows that the LC 50 animal studies of Carson, Eriksen, and Machle correlate quite well and ETL's and EEL's can be established at 0.6% and approximately 4% of the LC 50 values obtained for HF from animal experiments, respectively. However, fluorine ETL's and EEL's will probably be found to establish at levels of less than 2% of the LC 50 animal values.

(U) Other difficulties involve the reliability and accuracy of chemical methods or instruments available for determining when limits have been exceeded. Another problem is the number of repeat exposures and length of recovery periods in between which can be tolerated without encountering secondary pathological changes. This probably varies considerably among healthy normal individuals.

(U) It should be emphasized that there is sufficient research data (References 49 through 56) to fix the chronic tolerance levels for HF and F_2 at 6 and 1.7 mg/m³, respectively (3 ppm HF and 1 ppm F_2). Above these levels, the ability of the body to reject these quantities is jeopardized; fluorosis can be the long-term result.

(U) Another fluorine toxicity value which so far remains somewhat inviolate is the minimum lethal dose (MLD). Continuous exposure to 200 ppm of fluorine gas for 3 hours has proved fatal to human beings (Reference 64). An MLD set at 200 ppm-hours, therefore, appears to be a realistic figure. Currently established EEL's giving doses of 30 to 60 ppm-min. are quite conservative and provide better than a 300-fold safety factors under the MLD value.

(U) The following listing summarizes the toxic hazard data and knowledge important for establishment of guidelines on engineering design, range planning, operational constraints, personnel risk criteria on missile sites, and avoidance of legal risk arising from exposures of off-site population to fluorine oxidizers.

- a. The guideline EEL's established for the fluorine oxidizers as promulgated by NAS-NRC should be followed to control range personnel.

- b. Deviations should be allowed only after intensive evaluation by an informed specialist with the concurrence of the cognizant range safety or siting authority.
- c. In all cases, the general provisions of the Clean Air Act must be met.
- d. Instrumental analytical monitoring should be set up to confirm that the operational and safety procedures used to minimize toxic hazards both on-site and in adjacent uncontrolled populated areas are valid.
- e. Operations must provide the go-no go criteria based on the toxic hazard expected in case of a most credible incident which gives maximum assurance that dosages in uncontrolled areas do not exceed the ETL value (applicable only by permission of local authorities) or the TLV established for the particular oxidizer.

2. CLEAN AIR ACT

(U) The Clean Air Act is the basic federal instrument defining the procedures and controls imposed on organizations planning to use toxic propellants (Reference 65). The provisions of the act are administered through the U.S. Department of Health, Education and Welfare.

(U) In accordance with the requirements of the Act, range management and operating personnel are jointly responsible for conducting those studies necessary to provide clear-cut operational limits. Conformance requires (in part) the following procedures:

- a. Notify and work with local pollution control authorities (state, county and/or municipal control boards for air, soil, and water pollution).
- b. Provide pollution sampling and detection instrumentation for documenting peak and integrated pollution concentrations at the boundaries of the exclusion area and at facility boundaries.
- c. Select weather windows which will not permit pollutant drift of hazardous quantities downwind into populated area, taking all precautions to prevent exposure of humans, animals, and valuable plant life outside the exclusion area.
- d. Perform soil and water sampling and chemical analyses before and after tests to record and document possible pollution.

3. FACILITY REGULATIONS

(U) Facility regulation is of primary importance in order to provide adequate safety controls for the protection of employees, the population, plant and animal life, materials, equipment, and other property both on- and offsite.

(U) Failure analyses specifically instituted for a particular site and facility should be undertaken to achieve maximum safe operation. All the possible modes of failure must be anticipated so that correct engineering design and operation procedures may be employed to minimize unpredicted fluorine releases.

(U) Most credible release evaluations should be undertaken to design adequate facilities for disposal, purging, containment, and control of fluorine to atmosphere. Sources of fluorine or its principal reaction product, hydrogen fluoride, include the following:

- a. Ruptures of lines, diaphragms, valve seals, pumps, and quick disconnects.
- b. Rupture of storage or condensation tanks.
- c. Rupture of missile tanks during launch.
- d. Exhaust products from vehicle engines.
- e. Residuals from lines and other hardware after passivation, from parts being repaired or replaced, and from vent lines or parts under test.

(U) Range regulations at launch sites agreed upon by safety personnel of the cognizant agencies are set in each case in accordance with the provisions of the Clean Air Act and state or local air and water pollution laws and ordinances. Toxicity levels (EEL's), permissible emission rates, duration, and meteorological conditions must be evaluated in order to adequately locate (site) a toxic propellant operation.

(U) The responsibilities reserved by ETR safety authorities and those required of the range user for control of fluorine launch programs are abstracted (below) from Reference 66:

1. The basic philosophy applied to the determination of responsibility of conducting studies of the type described in referenced correspondence is: the Range will conduct those studies required to determine the capability of the Range to accommodate a program using hazardous material; the Range User is expected to conduct those studies necessary to assess a potential hazard to the Range resulting from the use of hazardous material.
2. Taking the foregoing into account, the following describes the studies and analyses required and the agency responsible for initiation of the studies.
 - a. Range Studies:
 - (1) Background Ecology. This study will determine existing levels of material from natural and/or artificial sources.

- (2) Routine Ecology Studies and Analysis. This will be done at intervals of time (depending on frequency of use of the hazardous material) to measure rate of increases when compared with initial background studies (Sub-Paragraph 2. a. (1) above).
- (3) The Range will analyze Range User studies of emission rates (see below).
- (4) The Range will conduct air sampling and sensing programs. This will be primarily "off complex" activity; however, it must be closely coordinated with "on complex" sensing, which will be done by the Range User (Sub paragraph 2. b. (5) below).
- (5) The Range will determine hazard radii and will develop and implement plans for the control of people.
- (6) The Range will, based on analysis of Range User Emission Rate Studies, establish propellant handling, transfer and launch operations restriction to those times when assured that hazardous material above the emergency tolerance limit does not exceed a boundary line two miles inside the Range boundary.
- (7) The Range will dispose of contaminated propellants.
- (8) The Range will analyze the vehicle propulsion system to determine validity of the Range User's Emission Rate Study. Complete schematics with stress specifications will be required.
- (9) The Range, through Medical Service people, will prescribe pre- and post-exposure medical examinations.
- (10) The Range will evaluate and approve, personal protective equipment and devices proposed for use by the Range User.
- (11) The Range will site a facility using fluorine to minimize influence on adjoining facilities, if appropriate. If an existing complex is converted to fluorine use, siting would not be appropriate.
- (12) Sampling and analysis will be accomplished by the Range. Maximum lead time prior to use of fluorine is required to allow procurement of necessary equipment. A minimum of six months is estimated to be required.

b. The Range User:

- (1) Conduct Emission Rate Studies. The complete propellant storage, transfer and loading system, as well as the vehicle, must be analyzed to determine likelihood of leaks and spills. The amount of material versus time versus meteorological conditions versus surface area and materials of construction must be considered to determine rounds per minute of vapor emitted. The launch abort following flight termination action or other incident must be included.
 - (2) Conduct studies to determine expected ecological effects.
 - (3) Determine methods of decontamination and neutralization.
 - (4) Determine composition of products of combustion and significant followon reactions.
 - (5) Maintain a sensor and repair crew on the complex while fluorine is on the complex. Portable sensors to scan components of the complete plumbing system must be used to discover leaks. A team of people qualified to operate the system to effect repairs and minimize loss of material will be required.
 - (6) Identify and propose for use, protective clothing, equipment and devices suitable for fluorine use.
 - (7) Plan and schedule use of the material to correspond with meteorological restrictions imposed by the Range.
 - (8) Determine emergency tolerance limits to which people may be exposed.
3. The foregoing attempts to describe work required of both the Range and the Range User. The list is not necessarily complete, but should serve as a guide to identify the total work required.
4. It should be noted that initiation of studies, scheduling of work, progress reports, etc., are dependent on a firm commitment to use fluorine. The location and amounts to be used, including back-up supplies, must also be identified."

4. STATE AND LOCAL REGULATIONS

(U) The California Standard of Ambient Air Quality, administered by the California Department of Public Health, Bureau of Air Sanitation, does not prescribe any legal limits on the quantity of fluorides or fluorine in the atmosphere. There is some discussion of undesirable concentrations (2 to 5 ppm of HF being regarded as causing skin and mucosa irritation), but these are not legal limits. Most counties in California with Air Pollution Control Boards follow the recommendations and limits determined by the Los Angeles County Air Pollution Control Board (LA APCB). There are no regulations of the LA APCB directly applicable to fluorine or fluorides. Rule 50 deals with making the air opaque by aerosols, smoke, and other means. The limit on opacity is 40%. Rule 52 states that particulate matter, including aerosols, shall not be emitted at a concentration of greater than 0.4 grain (mass)/cu ft of air. Total solid effluent allowed depends on the amount of material handled (Rule 53); for a throughput of 60,000 lb/hour, the legal maximum of solid effluent is 40 lb/hour. Data for Florida (ETR) are not at the moment available; air pollution in that state is under the control of Bureau of Sanitary Engineering, State Board of Health.

Appendix V

SUMMARY OF CURRENTLY USED QUICK DISCONNECTS

(U) A survey was made of existing quick disconnect (QD) couplings for possible adaptation to fluorine service. The specific couplings which have been reviewed are those used on Thor, Saturn IVB, Titan II, Centaur, and the APOLLO service module.

(U) Six-in. -diam couplings are utilized on the Thor. The ground half of the coupling is manually inserted into the vehicle half, an operation which also opens the vehicle poppet. No latching mechanism is used and decoupling as well as closing of the vehicle poppet is effected at liftoff by a combination of the forward motion to the vehicle and lateral movement of the ground half by an AGE actuator. The coupling is too large to be considered for the current fluorine QD program, and it utilizes soft seals incompatible with fluorine.

(U) Four-in. -diam QD couplings are used for loading propellants on the Saturn IVB. There are no shutoff valves in either half of the coupling. The coupling is manually installed and preloaded against a Teflon seal on the vehicle from a carrier plate which supports a majority of the umbilicals to the stage. Decoupling is accomplished at vehicle liftoff by pulling a release pin and moving the carrier plate clear of the vehicle with an AGE actuator. The coupling is too large to be used on the fluorine QD program, and the Teflon seal would not be incompatible with flowing fluorine.

(U) The 2-in. -diam Titan II coupling is in the proper size range to be a possible candidate for the fluorine QD application. It is designed for manual coupling and decoupling and has no quick release feature and no provisions for misalignment between the coupling halves. The coupling halves are joined by means of Acme threads. After initial engagement, manual rotation of the outer sleeve mates with the interface seal and further rotation opens the conical seat poppet valves in each half of the coupling to permit propellant transfer. Edge-welded metal bellows are utilized in the ground half of the coupling to provide positive sealing between moving parts and to provide a pressure-energized force on the interface seal during propellant transfer.

(U) The Titan II QD could not be readily modified to provide a quick release mechanism. The Teflon seals incompatible with fluorine under dynamic flow conditions (modification to metal seals would require major redesign), and proper cleaning of the metal bellows to ensure compatibility for fluorine service would be very difficult. Therefore, the Titan II QD is not suitable for this adaptation.



(U) Quick disconnect couplings of a 2 1/2-in. diam are used for loading propellants on the Centaur. Both the vehicle and ground halves of the couplings have shutoff valves controlled by the same AGE actuator. The valve in the vehicle half is spring-loaded closed and is opened by the opening motion of the valve in the ground half of the coupling.

(U) The QD is manually connected and held together by two frangible bolts which are designed to rupture within narrow applied-load tolerances. The interface seal is a U section, pressure-energized Teflon seal which utilizes a metal spring to maintain its shape prior to pressurization. Because of the low strength of the frangible bolts, the attaching nuts are torqued only fingertight.

(U) To prevent the formation of ice at the parting joint of the QD, a garter-type Mylar bag is installed across the separation flanges. The bag and joint are purged with helium to keep air and moisture away, and the bag slips off when the QD is decoupled.

(U) Disconnect of the QD is accomplished at liftoff. Lanyards are attached between the ground half of the QD and a retraction mechanism triggered by a liftoff switch. The two frangible bolts in the QD are ruptured by a suddenly applied load from the retraction mechanism through the lanyards, and the ground half of the QD and fill line are pulled free from the vehicle.

(U) The Centaur QD design is not suitable for use in fluorine service. The Teflon interface seal is incompatible with flowing fluorine and the use of low-rupture-strength attach bolts (low margin of safety for inadvertent loads) is not consistent with the required safety precautions for such a highly toxic and reactive fluid.

(U) A QD that is 2 1/2-in. in diameter is utilized in the Centaur H₂ vent system. The coupling is mated in a slightly recessed butt joint and locked together with two adjustable overcenter toggle latches. The interface seal is a sheared Teflon flat gasket and the housings are aluminum castings which form a 90° elbow when mated. The design pressure is 12 psig and permissible external leakage is 10 cc per hour, or approximately 10⁻² SCIM. The coupling must be manually engaged and adjusted to the desired preload; decoupling is accomplished by pulling a lanyard. The coupling contains no valves although it does have an orifice mounted in a swinging plate in the vehicle half which closes and effects a flow restriction when the QD is decoupled.

(U) Although some of the basic principles of this coupling might be utilized for the fluorine QD, the coupling would have to be redesigned to withstand a higher pressure and the interface seal would have to be changed to a material compatible with LF₂ under dynamic flow conditions.

(U) The Apollo service module utilizes 1-in. diam manually operated QD couplings similar in design to the Titan II couplings. They are too small for present fluorine application, and the connector is incompatible with fluorine for the same reasons discussed for the Titan II QD.

(U) Roylyn submitted a candidate QD for evaluation, a 2-in.-diam prototype model developed for a high-temperature pneumatic application. It was of all-metal construction, utilizing stainless steel for the body. The interface seal consisted of flat, precision-lapped surfaces with a machined Inconel X bellows to furnish the preloading force. A ball lock was used for latching mechanism and a lever-operated sliding sleeve served as a retainer for the balls in the latched position. The latch could be operated remotely by attaching a linear actuator to the lever. The AGE half of the QD contained a hinged shutoff valve actuated by the same lever that controls the latching mechanism.

(U) Although the QD was constructed of fluorine-compatible materials, there were several features in the design not acceptable for a QD for fluorine service. The interface seal on the vehicle half of the QD was vulnerable to damage during the mating operation. It is most unlikely that a suitably low leakage rate could be achieved with the type of seal used. The seal bellows would be very difficult to clean and inspect for cleanliness. Lap-type weld joints are used in the body and it would be impossible to determine that the lap joints were sufficiently clean for fluorine service. The latching mechanism is not adaptable for a redundant release system, a highly desirable feature for a QD for fluorine service.

(U) Concurrent with the survey of existing QD's a, survey of six vehicles (existing and study phase) was made to collect data on various weight, structural, and propellant parameters. The summarized data from this survey is presented in Table XXVIII to indicate some of the previous thinking regarding propellant loading requirements.

Table XXVIII
VEHICLE CLASSIFICATION

Vehicle	MANSAT	Saturn IV	Saturn IVB
Vehicle parameters			
Gross weight	20,000 lb	115,337 lb	245,000 lb
Vehicle type	Maneuverable, high energy upper stage	Booster second stage	Booster second/third stage
Propellants	LF ₂ /LH ₂	LO ₂ /LH ₂	LO ₂ /LH ₂
Oxidizer weight	14,615 lb	83,000 lb	184,000 lb
Oxidizer tank volume	162.8 ft ³	1262 ft ³	2828.6 ft ³
Oxidizer tank pressure	75 psia	46.5 psia	37-41 psia
Fill-line size	2" dia. (tentative)	3"	4"
Vent-line size	2" dia. (tentative)	5"	5"
PU System	Not determined	Closed loop (capacitance)	Closed loop (capacitance)
Structure			
Shell	Frame stiffened, corrugated skin	Integral with tanks	Integral with tanks
Oxidizer tank	Spherical, 81.5" dia. ~ 0.032 aluminum	200" dia., elliptical, common blkhd	Elliptical, common blkhd
Insulation	High performance on tanks (ground purge)	Interior of tk. and in common blkhd	Interior of tank and common blkhd
Umbilical			
Oxidizer	LF ₂ vent, LF ₂ fill/drain	LO ₂ fill/drain	LO ₂ fill/drain
Fuel	LH ₂ vent, LH ₂ fill/drain	LH ₂ vent, LH ₂ fill/drain	LH ₂ vent, LH ₂ fill drain
Propellant loading parameters			
Oxidizer flow rate	Not determined	(Total fill time = 20 min.)	Approx 30 min.
Hold time	No known limit	7 min. max. while pressurized	3 min. while pressurized
Oxidizer tank pressure	Near ambient during fill	48.5 psia (vent)	44 psia (vent)
Oxidizer condition	Possible slight subcool (-307 F; -315 F)	Boiling point at ambient press.	Boiling point at ambient press.
Remarks		Final topping while pressurized Vehicle Q. D. and line are drained and purged prior to liftoff. Disconnect by actuator at liftoff.	Final topping while pressurized. Vehicle Q. D. and lines are drained and purged just prior to liftoff.
Reference	67	68	69

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Table XXVIII

CLASSIFICATION (U)

IVB	Centaur	Delta	Nomad (G-1 Early Flight Vehicle)
14,000 lb	32,000 lb	14,000 lb	14,300 lb
Third stage	High energy upper stage	Second Stage Booster	Feasibility test: second stage
LO ₂ /LH ₂	LO ₂ /LH ₂	IRFNA/UDMH	LF ₂ /Hydrazine
14,000 lb	23,400 lb	8,000 lb	5,300 lb
16.6 ft ³	407 ft ³	62 ft ³	79 ft ³
31 psia	32 psia	340 psia	275 psia
	2.5 in.	3/4"	1.0" dia.
	2.5 in.	1/2" (overflow and vent)	1/2" dia. (emergency vent)
Resistance)	Closed loop (ΔP.)	Open loop	Open loop
	Integral with tanks	Integral with tanks	1/8" alum. cylinder with stiffeners
on blkhhd	10' dia., elliptical	55" dia., cylindrical, common blkhhd	61.5" dia. alum. sphere with LN ₂ cap
and common blkhhd	Plastic, exterior, jettisonable	None	Fiberglas external tank insulation
	Not determined	Manually connected MS fittings	F ₂ fill/drain, F ₂ press/vent
fill drain	LH ₂ vent, LH ₂ fill/drain	Fill and drain ports capped after fill	Hydrazine fill/drain
Pressurized	500 GPM (max.)	20 GPM	Not determined
	2.1 min. after topping	Launch 1 day after fill	Not determined
Ambient press.	29 psia	Ambient	250 psia
Be pressurized.	Boil off for 20 min. in veh. tank	Ambient	Subcooled to -320 F
	External leakage = 5 scfm max.	Propellant loading valves are hand operated.	Top off before pressurizing
lines are drained prior to liftoff.	For all couplings at interface vehicle - half valve (fill and drain)	Fill tanks until liquid comes out the overflow and then drain "x" lbs.	Oxidizer tank has a high press. relief system separate from the pressurization/vent system and Q.D.
	Both ox and LF ₂ = 500 scfm.		
	70, 71, 72, and 73	74	75 (C)

Appendix VI
SUMMARY OF LF_2 QD¹ AND RELATED STUDIES

1. INTRODUCTION

(U) A review of the available literature on fluorine research and experimentation indicates that there have been very few attempts to define or build a QD for fluorine service. In one instance, a QD was designed, built, and tested by NASA at the Lewis Research Center (LeRC), Cleveland, Ohio. A preliminary design concept for an LF_2 QD has been suggested by Aerojet-General Corporation and is presented in this section.

(U) The present work by Douglas was initially aimed at meeting a set of requirements for an LF_2 QD defined by the Request for Proposal, Reference 76. A preliminary concept to meet these requirements was evolved and is presented herein.

2. NASA LeRC QD

(U) A prototype QD coupling for ground to vehicle transfer of LF_2 or FLOX was designed, fabricated, and demonstrated at LeRC in 1963 (Reference 77). The primary features of this QD coupling include remote separation capabilities and negligible F_2 spillage when disconnected.

(U) Each half of the coupling has its own poppet-type shutoff valve spring-loaded to the closed position. The interface surfaces are basically plane surfaces with deep intermeshing serrations which serve to align the two halves during mating and also form a labyrinth path between the inner and outer interface seals. Two metal V-ring seals located concentrically on the separation interface and with vertex of each V oriented outward are used to prevent external leakage. The concept for sealing is that any leakage past the inner V-seal will gasify between the labyrinth faces from normal heat transfer, thereby building up pressure in the interseal cavity and causing the gas to leak back into the liquid flow passage, since the orientation of the V-seals offers less resistance to leakage in the inward direction.

(U) Latching is accomplished by three mechanical fingers which pivot on the AGE half coupling and lock over a flange on the vehicle half coupling. Pneumatic cylinders were used to operate the latches and move the AGE half coupling in and out of engagement. The coupling valving is opened

by pressurizing an integral pneumatic cylinder in the AGE half coupling which opens both poppet valves simultaneously. When the pneumatic pressure is vented, the valve springs return the poppets to their respective seats.

(U) The coupling was subjected to a limited amount of functional testing and operation was reported satisfactory within the range of imposed test conditions.

3. QD CONCEPT FROM LF₂ AGE HANDBOOK

(U) General design approaches for both insulated and uninsulated LF₂ QD couplings are presented in Reference 78. The concepts are similar, except that overlapping vacuum-jacketed areas are proposed for the insulated concept.

(U) The design has shutoff valves in each half and both coupling and decoupling are manual operations. Soft metal is proposed for the interface seal, which is accomplished by bringing the two halves of the coupling together and latching with a ball lock mechanism. An interlocking pin prevents opening the valves before the coupling halves are engaged and locked together.

(U) An outer sleeve on the AGE half housing is rotated on a coarse screw thread to open the valve poppets. A conical bellows seal permits axial motion to be transmitted to the poppets and prevents leakage past the operating mechanism.

(U) A positive interlock is provided so that the coupling cannot be separated when the valves are open. Stainless steel conical poppets with soft copper or aluminum seats are proposed.

(U) The design aim for the coupling is to prevent any spillage, or to permit only a small wetted area on the coupling halves when separated. A simple enclosure built around the disconnect and supplied with an inert gas purge was proposed for dissipating any F₂ remaining on the interface between the coupling halves.

4. ORIGINAL DOUGLAS CONCEPT

(U) The first concept considered by Douglas was based on a set of design requirements which were defined by the Request for Quotation, Reference 76. The significant features of this concept were as follows:

- a. Remote coupling and decoupling.
- b. Shutoff valves in both halves of the coupling.
- c. Misalignment capability.

- d. Double seat at interface.
- e. Purged interface seal cavities.
- f. Redundant release mechanism.

(U) Figure 42 is a sketch of the concept for the vehicle half of the coupling. It consists of a valve body with a spherical outer shape for an interface with the AGE half of the coupling. The shutoff valve in the vehicle half coupling consists of a spring-loaded spherical poppet which closes against a narrow land seat formed by the intersection of two spherical concave surfaces, to produce a perfectly circular seat. The desired seat width would be formed by a combination of coining with a hard tooling ball and lapping with a spherical lap.

(U) Figure 43 is a sketch of the two halves of the coupling connected. The latching mechanism consists of a number of balls which are locked into the latched position by a spring-loaded outer sleeve. Unlatching is achieved by applying pneumatic pressure to Port A; this retracts the sleeve and permits the balls to move outward and release the vehicle half coupling. The shutoff valve in the AGE half of the coupling has a spring-loaded spherical poppet similar to but smaller than the vehicle half. The AGE poppet is connected by a shaft to a pneumatic cylinder. Application of pneumatic pressure to Port B opens the AGE valve which in turn pushes the vehicle valve open. The valves are closed by their respective springs when the pressure is vented through Port B.

(U) The spherical interface between the coupling halves permit 2° misalignment in any direction between the vehicle and AGE equipment. A concept for a primary interface seal is shown in Figure 44. This concept consists of two thin lips machined into a replaceable metal ring. A secondary seal just outside the primary seal is shown in Figure 43. It was planned to use Teflon for the secondary seal because it would only be exposed to a dilute mixture of gaseous fluorine leaking by the primary seal and N₂ or He purge applied to Port C and exhausted through Port D.

(U) A redundant disconnect mode is effected by cutting a number of slots in the outer (ball retention) sleeve and adding a thin metal ring to reinforce the slotted member during normal operations. A lanyard connected to this outer sleeve would provide sufficient force to rupture the reinforcing ring and thereby release the locking balls in the event of failure of the primary release.

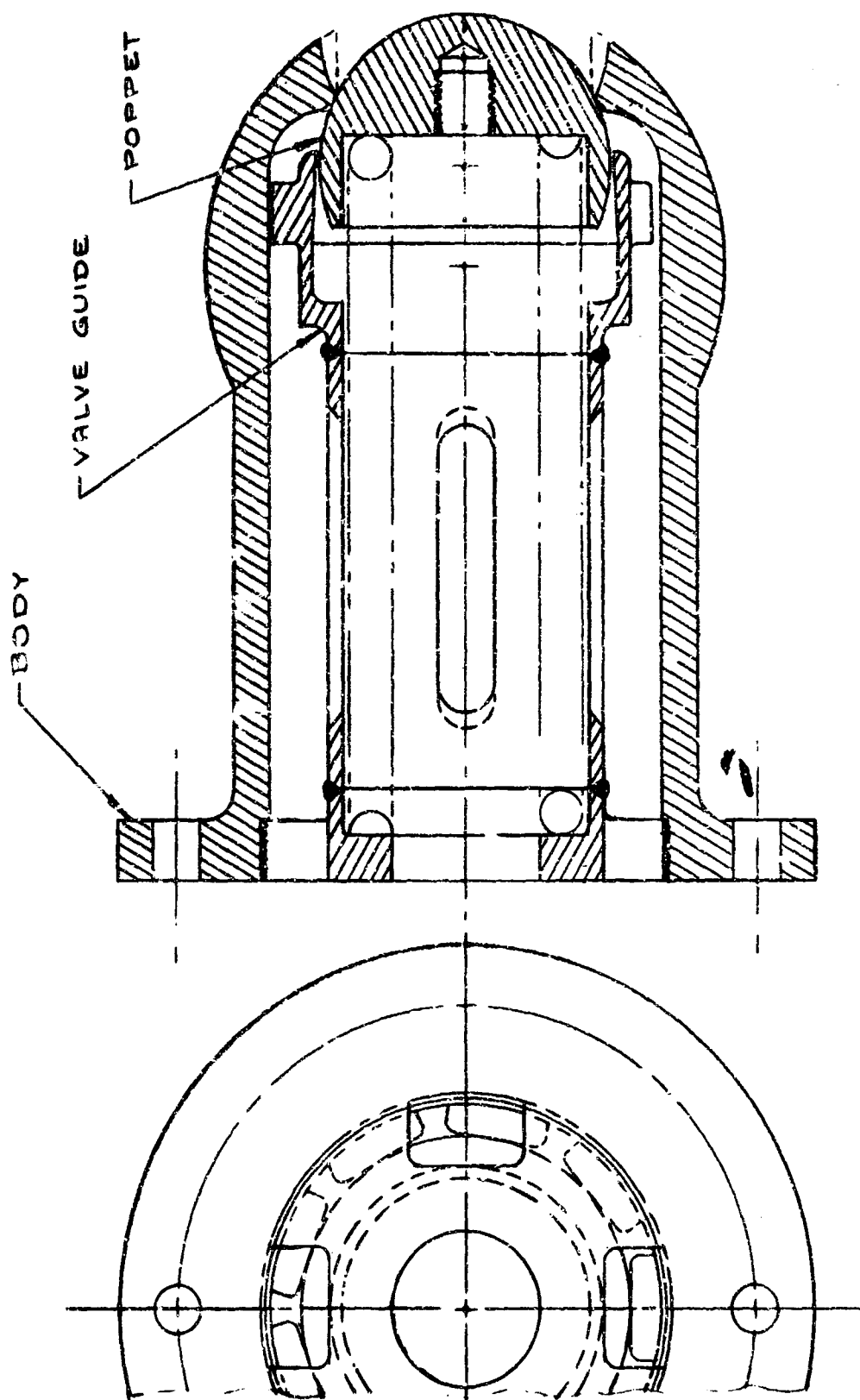


Figure 42. Vehicle-Half of Quick Disconnect

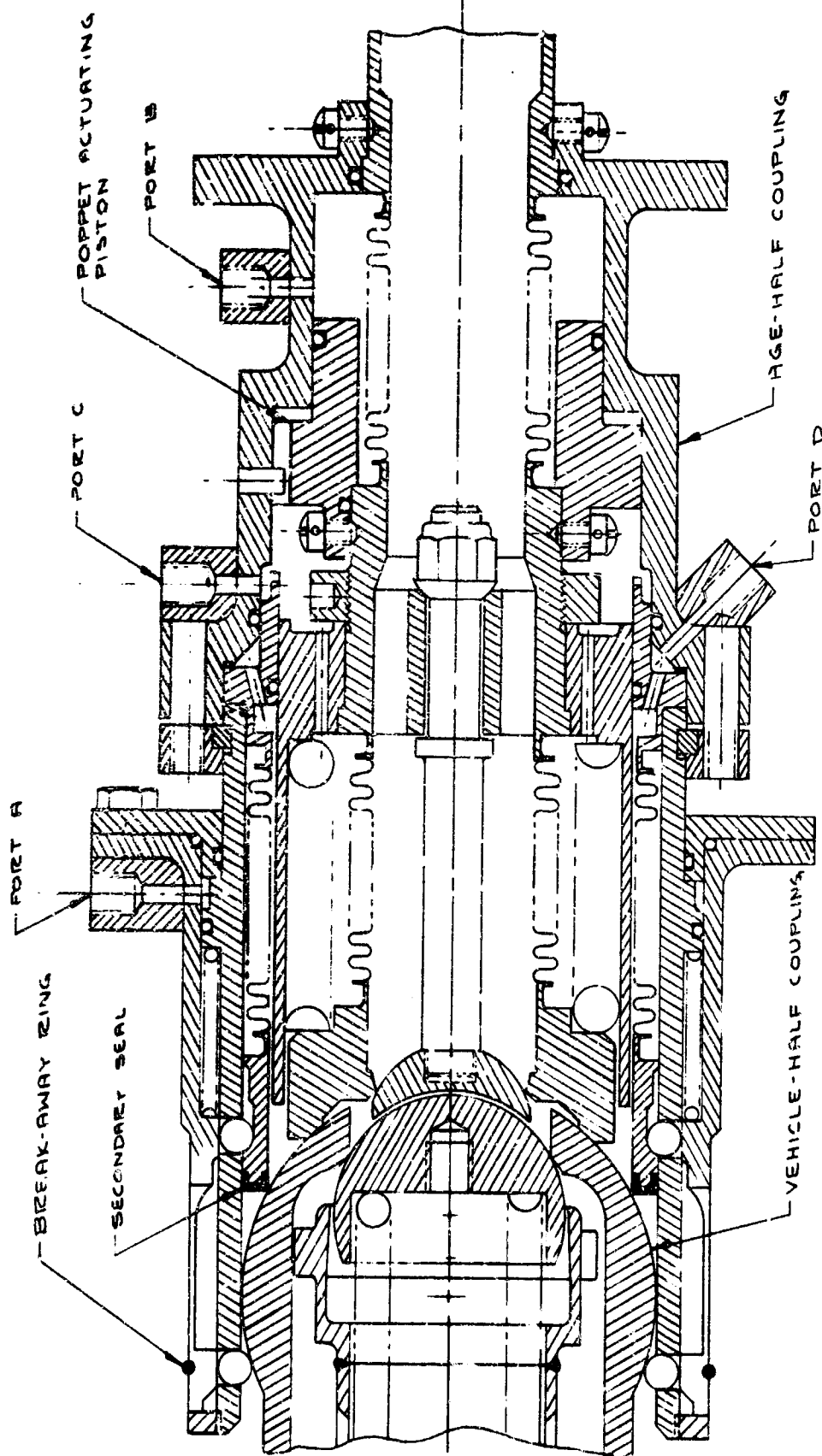


Figure 43. Quick Disconnect, Latched and Valves Closed

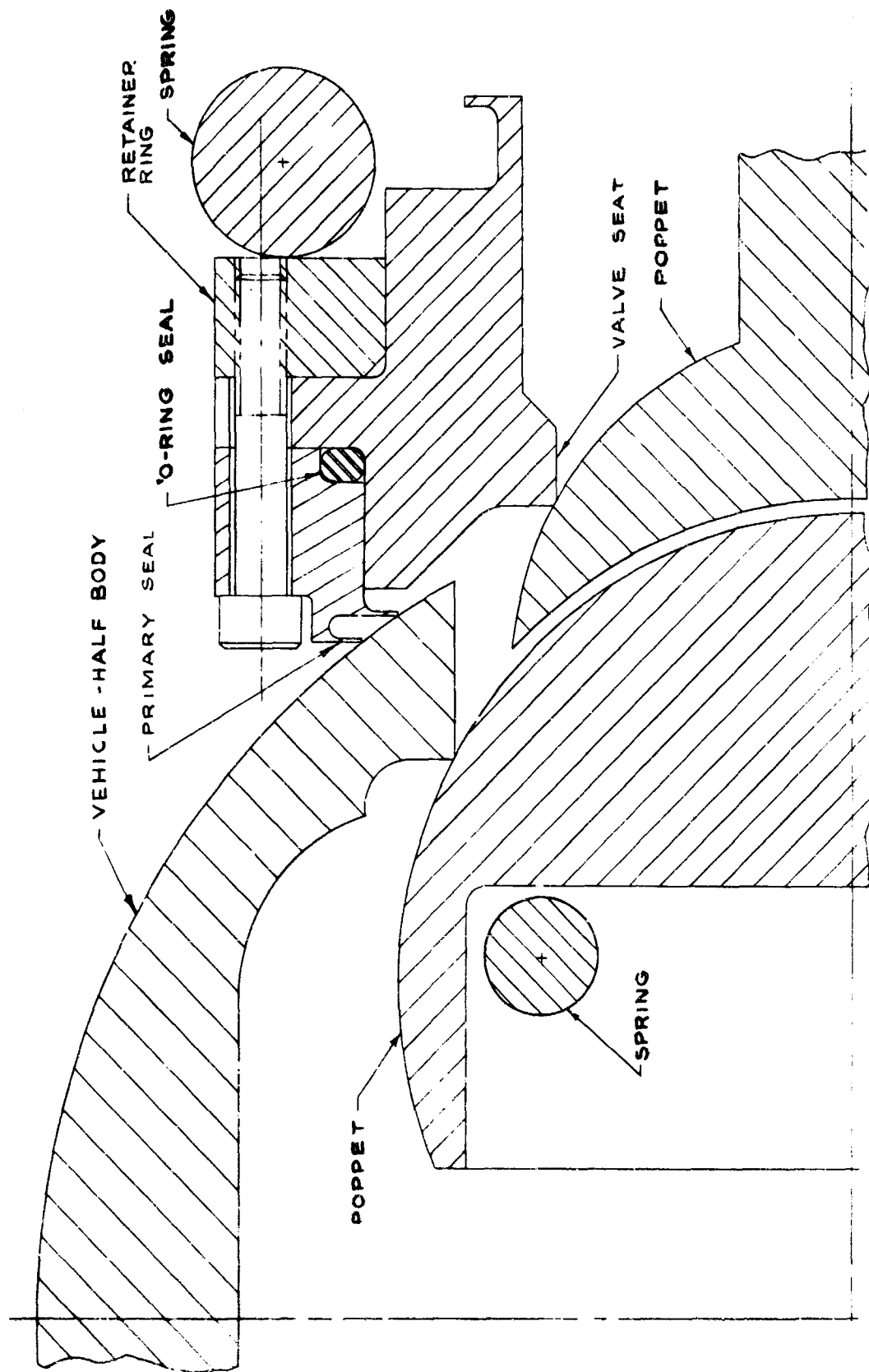


Figure 44. Interface Seal Concept

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13. ABSTRACT <p>The Phase I effort described here has resulted in a set of preliminary criteria for quick disconnect (QD) couplings for use with fluorine and fluorine containing oxidizers. For this study a number of vehicle systems were examined to determine the fundamental requirements for couplings. Also, data were obtained from literature, government and industry contacts, and Douglas studies. Identified are the requirements imposed on a typical upper stage oxidizer fill-and-drain QD by vehicle and AGE considerations, disconnect technology capabilities, specific information voids identified during this study, and program plans for Phases II and III.</p> <p>The criteria established suggest that the design, fabrication, and testing of a fluorine QD can be undertaken with great confidence that the resulting product will serve its intended purpose.</p> <p>Phases II and III provide for demonstration of these criteria by fabrication and testing of two fluorine test model QD's, establishment of a preliminary military specification for a fluorine QD, and delivery of a prototype fluorine QD.</p>			

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Security Classification

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Security Classification

14. KEY WORDS		LINK A		LINK B		LINK C	
		ROLE	WT	ROLE	WT	ROLE	WT
Fluorine	Materials						
Chlorine trifluoride	Fabrication						
FLOX	Seal Analysis						
Compound A	Latches						
Quick Disconnect Coupling	Contamination						
Seals	Cleaning						
Leakage	Failure Modes						
Toxicity	Purging						
Leak Detection							

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